Naval Surface Warfare Center
Carderock Division
Bethesda, MD 20084-5000

NSWCCARDIV-TR-96/017 July 1996

Survivability, Structures, and Materials Directorate

Technical Report

Practical Guidelines for Moisture Diffusion Measurements in Composites

by

Joseph M. Augl

19961008 190

REPORT DOCUMENTATION PAGE		Form Approved OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE July 1996	3. REPORT TYPE AND DATES COVERED Interim; 1 January 1996 - 1 July 1996

4. TITLE AND SUBTITLE Practical Guidelines for Moisture Diffusion Measurements in Composites	5. FUNDING NUMBERS SC2B; RS34S56
6. AUTHOR(S) Joseph M. Augl	

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Naval Surface Warfare Center Carderock Division White Oak Laboratory 10901 New Hampshire Ave. Silver Spring, MD 20903-5640	8. PERFORMING ORGANIZATION REPORT NUMBER NSWCCARDIV-TR-96/017

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Office of Naval Research Ballston Tower One 800 N Quincy St Arlington, VA 22217-5660	10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.	12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

Composite powder samples and thin sheet laminates were investigated for their moisture absorption behavior. Knowledge of the maximum moisture equilibrium concentrations in composites as a function of the surrounding relative humidity is necessary for determining their moisture diffusion coefficients. While thin sheet laminates gave the same solubility values as thick laminates, powder samples of the same laminates gave about 25 percent higher values. These apparently higher solubilites in composite powder samples were attributed to the larger surface area per unit mass. Based on these findings, procedural guidelines were proposed for increasing the efficiency in determining the diffusion coefficients in composite materials.

14. SUBJECT TERMS moisture diffusion composite materials diffusion coefficient	15. NUMBER OF PAGES 69
	16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT SAR

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102

CONTENTS

ILLUSTRATIONS

ILLUSTRATIONS (continued)

ILLUSTRATIONS (continued)

TABLES

ADMINISTRATIVE INFORMATION

This project investigates the aspects of moisture sorption and diffusion as a function of time, temperature and humidity. This report is one of several that document the project. It provides guidelines to scientists and engineers who model composite materials property changes in marine environments. The purpose of this report is to reduce the experimental time for obtaining the diffusion coefficients and sorption behavior of composite laminates and sandwich materials without sacrificing accuracy.

INTRODUCTION

Prediction of life cycle costs for maintenance of new Navy hardware is very important in the acquisition process and must be introduced in the early stages of the engineering development. Long-term effects of the environment will change the initial properties of the materials used in these structures. In order to simulate the property changes of what might be considered the worst case scenario, structural test items are usually exposed to an accelerated environmental condition thought to represent desired conditions. This approach is often crude and misleading. Using available computational tools, such as desktop computers, one can obtain better results at greatly reduced cost. This includes better estimates for accelerated times and temperature conditions in polymeric and composite materials exposed to real marine environments.

Strength, stiffness, stress relaxation, creep, shear deformation, and other matrix dominated properties of organic polymers and composites depend on the local concentration of absorbed moisture and on the local state of stress. These relations must be determined independently. The local moisture concentration, however, can be predicted with a high degree of reliability, and is an important first step in modeling these property changes. The tools for these diffusion analyses are finite difference or finite element codes, which we discussed in other reports.[1,2]

The purpose of this work is to provide the materials scientist and engineer with guidelines for performing the necessary experiments to obtain moisture diffusion coefficients and solubilities in polymers or composites. The detailed suggestions, given in this report, may be obvious to those experienced in this field, but those who intend to get fast results, without having to engage the expert or to go through a long learning curve, may profit from it. The main objective is to provide suggestions for reducing the times required for these measurements to a minimum. Our discussion will be limited to procedures which require no more than a semimicro-analytical balance, a series of desiccators for providing constant relative humidifies (RH), and ovens that can be controlled to $\pm1.0°C$; in other words, a standard laboratory environment with no more than $\pm1.0°C$ temperature fluctuation.

One of the simplest methods (discussed in this report) for obtaining moisture diffusion coefficients is to expose pre-dried plate samples to different constant RH's and measure the weight gain with an analytical balance. The diffusion coefficients are then derived from the initial weight gains. This approach is described in many papers and in Crank's "Mathematics of Diffusion."[3] Although the analysis of this method is straight forward, it usually takes a long time to complete the experiments. For instance, if one uses plate samples with 0.1-inch thickness, it may take from 6 months to one year at ambient temperatures to get the samples to an equilibrium concentration with the specified RH. On the other hand, if the samples are too thin, one can easily make a significant error in the initial slope of the moisture uptake. Therefore, we recommend using several sets of different laminate thicknesses for the experiments. We found it necessary to discuss a

number of experimental issues which may affect the accuracy of the diffusion and sorption measurements, especially, if one wants to reduce the measurement times to a minimum. To demonstrate the validity of the arguments made for shorter measurement procedures, we compared our suggested approach with previous results obtained from single sets of specimens. While the number of specimens to be machined for this purpose is larger, the overall time to get the desired results is significantly shorter and therefore less expensive.

EXPERIMENTAL

COMPOSITE MATERIALS

Three composite laminates, two E-glass/vinyl-ester laminates and one E-glass/epoxy laminate were provided by C. Williams of NSWC Carderock Division, Annapolis Site. The first two laminates were nominally 0.1 inch thick composites made from 24 oz. E-glass woven roving /510A Dow Derakane vinyl ester fabricated using VARTM. These two laminates had the same formulation, yet they showed slightly different maximum moisture uptake and slightly different diffusion coefficients[4,5]. Both laminates will be called RTM3 for brevity sake. The first laminate was made as an individual panel and will be called RTM3 (Fab. 1) the second panel was removed from the undamaged part of an impacted sandwich panel which had a 1 inch PVC foam core. The third composite was an E-glass/epoxy(G10) laminate (reported in reference 4)

MAXIMUM MOISTURE EQUILIBRIUM CONCENTRATION

Powder Samples.

A 7/32 inch diameter drill bit was used to drill holes into the composite sheets. About one gram of turnings were used per powder sample. The powder samples were placed into a small porcelain crucible, nominally of four gram weight and one inch maximum diameter. The samples were dried over drierite or equilibrated in an environmental chamber before weighing. The time when the environmental chamber was opened, was defined as time zero. The sample was placed on a magnetically damped semi-micro balance. The first weighing was done one minute after removing the sample from the environmental chamber. The sample weight gain (or loss) was recorded in intervals of one minute for the next 5 to 10 minutes. Note that the RH in the laboratory is usually different from that in the environmental chamber (higher or lower); therefore, the equilibrium moisture concentration can only be determined from the zero time extrapolated differences between the weights of the totally dry samples and their weights after equilibration at a specified RH. If more than one sample was in the environmental chamber, their removal times were spaced 3 hours apart to ensure equilibrium inside the desiccator.* This is especially important for powder samples because they sorb very rapidly. Every time the chamber is opened for sample removal, the equilibrium RH in its interior is disturbed. Since there is no exact theory of moisture uptake for a powder with an unknown distribution of particle size, we defined the zero weight as the linear extrapolation of their weight changes to zero time. The samples had attained equilibrium with their environments after one or two hours (laboratory or environmental chamber).

---

* The terms desiccator and environmental chamber are used here interchangeably for both drying the specimens and exposing them to specified relative humidities. Humidity is applied by using various salt solutions in separate containers at the bottom of the desiccator. Remaining undissolved salt crystals keep the RH constant.

Thin Sheet Composite Plate Samples

The composite laminates which, were usually 1/8 to 1/10 inch thick, were used for machining specimens with 0.010 to 0.016 inch thickness. Before the specimens were machined to their final thickness, both surfaces were machined off to ensure that any resin reach surface area was removed. The composite laminates were then mounted with a double-stick tape onto a grinding bench with a parallel surface grinding wheel to give a uniform thickness. After machining, the samples were carefully removed from the surface of the grinding bench with a wide, flat spatula. The thickness of each sample was measured at 9 different places and averaged. The moisture equilibrium concentrations were again defined from the difference of their zero point extrapolations between dry and moisture equilibrated specimens. The initial weight gain for flat plates is proportional to the square root of time. Therefore, we plotted the square root of time versus weight as an extrapolation procedure to obtain zero time weights. The weight gains for the thin sheet samples were considerably slower than for the powder samples, therefore one obtained a better extrapolation. One can use the first 4 to 8 points for the extrapolation to zero time.

ENVIRONMENTAL CONDITIONING

Glass desiccators were used, for both drying and RH equilibration. Saturated salt solutions with undissolved excess salts were used for establishing specific RH's (potassium chloride for 80 percent RH, and sodium acetate for 23 percent RH). The salt solutions were placed in a wide dish at the bottom of the desiccator and the samples were placed on sample holders above the dividing, perforated porcelain plate. In this investigation, we made only room temperature measurements. If one intends to make measurements at elevated temperatures, one needs to modify the procedures somewhat in order to accommodated the faster sorption rates. One needs to choose sample thicknesses that will reduce the errors of the measurement. A rapid temperature quenching to ambient weighing temperature is necessary. This may be accomplished by placing the plate samples between two metal blocks that are at ambient temperature.

MEASUREMENT OF DIFFUSION COEFFICIENT ON THICK SPECIMENS

While the measurements of the maximum moisture solubilities in composites should be done on thin plates, it is recommended that the diffusion coefficients which is determined from the initial sorptioin curves be determined on thick plate samples. The reason for this recommendation is accuracy, and will be discussed in the following sections of this report.

DISCUSSION

DETERMINATION OF THE MOISTURE DIFFUSION COEFFICIENT BY THE SORPTION METHOD

The simplest method for determining the diffusion coefficients in composites and probably the most accurate, is based on the solution of the Fick's diffusion equation for an infinite plate geometry which is given in Equation (1), (see reference 3, p.48).

$$\frac{M_t}{M_\infty} = 1 - \sum_{n=0}^{\infty} \frac{8}{(2n+1)^2 \pi^2} e^{-D(2n+1)^2 \pi^2 t / 4l^2} \tag{1}$$

where M_t is the amount of moisture absorbed after time t, and M_∞ is the maximum amount of moisture that can be absorbed for a given outside RH after an infinite exposure time, D is the diffusion coefficient, and l is 1/2 the plate thickness. On plotting M_t/M_∞ versus $(Dt/thickness^2)^{1/2}$ one obtains a dimensionless graph (see Figure 1, solid line) which is the same for all materials obeying Fickean diffusion. For the calculation of the graph in Figure 1, we used the first 20 terms of the infinite series of Equation (1). The initial slope of the curve is a straight line up to a value of about 0.5. Thus, on plotting the experimental sorption data as M_t/M_∞ versus $(t/thickness^2)^{1/2}$, one can derive the diffusion coefficient from the slope of the initial part of the sorption curve where the value of M_t/M_∞ is about 0.5, (see reference 2, p. 239; note in the reference l is called the thickness while in Equation (1), it is half the thickness). After simplifying and rearranging Equation (1) the average diffusion coefficients are obtained from Equation (2).

$$D = (\pi/16) S^2 \tag{2}$$

where $S = d(M_t/M_\infty)/d(t/(2l)^2)^{1/2}$ is the initial slope of the curve.

In a recent technical report (reference 4), we have described the determination of diffusion coefficients of several materials using this approach. A major shortcoming of the experimental work we described therein was that it took about one-half year to determine the diffusion coefficients of composites at room temperature. Once M_∞ is known, it takes no more than one or two weeks to get sufficient data for the initial slope of the sorption curve from reasonably thick specimens (0.09 inch). However, it takes more than ten times as long to ensure that there is no further weight gain, and that at least 99 percent of the equilibrium concentration has been reached. Since one needs to know the maximum equilibrium concentration (M_∞) for a given RH in order to calculate D from equation (2), it was desirable to develop a method that would shorten the time to determine M_∞ without sacrificing accuracy of the measurement.

5

From inspection of the dimensionless graph of Figure 1 we can get other important information. We see that more than 99 percent of saturation for a given RH is attained when the condition of Equation (3) is satisfied.

$$(Dt/l^2)^{1/2} = 1.4 \qquad (3)$$

Solving Equation (3) for t when D and the plate thickness is known, one can estimate how long it takes to attain maximum moisture saturation. Or vice versa, how thin the specimen must be in order to saturate it in a given time. At $M_t/M_\infty = 0.5$ one obtains from Equation (1) a $(Dt/l^2)^{1/2}$ value of 0.4436. Solving for t, one gets Equation (4)

$$t = 0.4436^2 \, l^2/D \qquad (4)$$

This expression gives the same result as that obtained in reference(3 p. 239), where the diffusion coefficient can be obtained from Equation (5)

$$D = 0.049/(t/th^2) \qquad (5)$$

where the time (t) is taken when $M_t/M_\infty = 0.5$, $th = 2l$ is the sample thickness.

Equations (3) and (4 or 5) indicate that it takes about 10 times as long to fully saturate the material with moisture than to sorb only 1/2 the amount. Therefore, it would be desirable to obtain the maximum moisture concentration, M_∞, for a specific constant temperature and RH value, from an independent experiment that does not take longer than measuring the initial slope with reasonable accuracy or to interpolate the time when M_t/M_∞ is 0.5. This is one of the objectives of this work.

From what we have said above, it seemed reasonable to use very thin sheets of composite or even powdered samples to attain a very short saturation time and use the measured saturation value, M_∞, and the short-time M_t values from thicker plate samples, for the calculation of the diffusion coefficient. To use only thin sheet composite samples for measuring the diffusion coefficients may lead to unacceptably large errors which we will discuss in the next section.

For an accurate environmental model, one also needs to determine the change of the diffusion coefficient with temperature and concentration. The change of the diffusion coefficient with concentration can be measured simultaneously by using separate desiccators with different saturated salt solutions inside the desiccators that provide different constant RH values. The samples are exposed in these atmospheres of constant RH. For most polymers, one finds that Henry's Law (that is, the sorbed amount is linearly proportional to the partial pressure in the gas phase) applies only approximately. The Flory-Huggins or the Brunauer, Emmett, and Teller sorption isotherm may be more

applicable. However, for most polymer matrix composites, the curvature is small and may be approximated with a simple second or third order least square fit of the sorption data sufficient to represent the relation of RH to the equilibrium concentration.

Finally, we have to consider the change of the diffusion coefficient with temperature. Again, in order to optimize the sample thickness for these measurements at elevated temperatures, it is prudent to estimate a suitable sample thicknesses for the temperatures of interest. This can be done once a diffusion coefficient has been measured for some reference temperature. Since diffusion is a thermally activated process, the diffusion coefficient follows an Arrhenius behavior that can be represented by Equation (6)[6]

$$D = D_o \exp(-E_{D,0}/RT)\, f(c) \tag{6}$$

where $E_{D,0}$ is the activation energy of diffusion, R is the ideal gas constant, T is the absolute Temperature in °K, f(c) is the exponent(αc), c is the concentration, α is a temperature dependent constant and D_o is a constant. For a small temperature range a mean activation energy E_D can be defined by the simpler Equation (7) (see Reference 6)

$$D = D_o \exp(-E_D/RT) \tag{7}$$

or equivalently by Equation (8)

$$\ln(D) = -E_D/RT + \ln(D_o) \tag{8}$$

A plot of ln(D) versus 1/T is the equation of a straight line. From its slope one obtains the activation energy of the diffusion of moisture in composites, which, from our experience, was found to be in the range of 9 to 14 kcal/mole. For a known diffusion coefficient at some reference temperature, and by taking an average activation energy of 12 kcal/mole, one can use equation (8) to make a rough estimate for the diffusion coefficient at some elevated temperature. This will help in deciding the sample thickness for the elevated temperature experiments (using equations (4 or 5)). Figure 2 shows an example where the diffusion coefficient at 23°C was given as 8.39E-09 cm^2/sec. The corresponding Arrhenius plots for 10,11,12,13, and 14 kcal/mole are shown. The expected diffusion coefficients for these activation energies at 35, 50, and 60°C are located on the ordinates of the abscissa values 3.245, 3.095, and 3.002 respectively.

For the environmental model, we find that we need a three-dimensional surface to describe the diffusion coefficient as a function of temperature and surface RH, or equivalently, of the corresponding moisture concentration in equilibrium with RH.

Since we have assumed that the change of D with temperature, when plotted according to Equation (7), is a straight line, and since the change of D versus

concentration is a smooth curve that can be described with a simple second or third order polynomial, we propose to use the following semi-empirical Equation (9)

$$z(x,y) = a_{12}(x - x_1) + b_{11}(y - y_1) + b_{12}(x - x_1)(y - y_1)$$
$$+ c_{11}(y - y_1)^2 + c_{12}(x - x_1)(y - y_1)^2 + z_1 \qquad (9)$$

for describing the moisture diffusion coefficient as a function of temperature and concentration. For simplicity, we have used Cartesian coordinates, were z represents the natural (or the common) logarithm of the average diffusion coefficient, ln(D); x represents $1000*1/T$ (in °K); and y represents the RH value at the surface of the specimen or the respective maximum moisture concentration of the composite in equilibrium with the RH value. The latter is obtained experimentally. From the measured values of diffusion coefficients as a function of temperature and concentration, the unknown parameters a_{12}, b_{11}, b_{12}, b_{22}, c_{11}, and c_{12} are determined by an iterative process (a three-dimensional, nonlinear, least square fitting routine according to an algorithm by Marquardt-Levenberg). One of the centrally located diffusion coefficients is taken as a reference point, (x_1,y_1,z_1).

Knowledge of solubilities and diffusion coefficients as a function of RH and temperature are basic requirements for environmental modeling. In this report we don't intend to discuss how these data are to be used to model the environment and the transport of moisture in composite structures. Suffice it to say that such a model requires both, a mathematical presentation of the environment giving the boudary conditions of the model, and the moisture transport behavior of the material, that is, the diffusion coefficient as a function of temperature and concentration. Finite element or finite difference methods are usually employed for this purpose. Here we will only provide experimental guidelines for making diffusion coefficient measurements more efficient.

EXPERIMENTAL CONSIDERATIONS

For high quality composites, that is, composites with low void content, one could calculate their diffusion coefficients from the fiber volume fraction and from the resin diffusion coefficients as we have shown previously.[7] For equal thicknesses of resin and composite plate specimens, it would take only about one fifth of the time to measure the resin diffusion coefficient. Since most composites have voids of various kinds, which affect their diffusion coefficients, we recommend measurement of the diffusion coefficient for composites directly. Other methods for measuring diffusion coefficients, which are based on measuring changes in volume or partial pressure of moisture or where a Cahn electric micro-balance is placed inside a vacuum manifold will not be discussed here.

The procedure for obtaining the moisture diffusion coefficient described here is simple and reasonably accurate, yet, it requires careful analysis of the various steps and attention to details which we will discuss below.

Weighing Errors

The semimicro-analytical Mettler balance used for the sorption measurements has an accuracy of ±0.00001 grams (not counting possible drifts in calibration due to changes

in temperature or RH in the laboratory atmosphere). With a maximum moisture solubility of 0.17 percent at 80 percent RH and for a 1.0 gram powder sample (which was the nominal amount of powder used for the measurements) this results in a 0.00170 grams difference between the dry and fully moisture saturated samples. Considering the weighing errors of the crucibles which contains the powder samples, one may expect at least an error of ±2 percent in the weight gain measurements with powder samples.

For thin plate samples with a nominal thickness of 0.010 to 0.016 inch and a size of 2 x 2 inches, the resulting weights where about 1.5 grams which improved the accuracy slightly to about ±1.5 percent. Also, the extrapolation procedure for the zero time weights is probably more reliable since it is based on a theoretically defined sorption process.

In the past, we used a Cahn-Electro-Balance within a vacuum manifold to follow automatically the moisture sorption. This method has some advantages when one needs to measure only very few specimens, but one should not expect a higher accuracy of the results, even though the sensitivity of the Cahn-Electro-Balance is an order of magnitude better (10^{-6} grams) than the semi-micro balance. However, since the sample size for the Cahn balance was very small (in the order of 0.1 gram), there was no advantage in using it. It suffers the same problems, that are inherent with measuring thin specimens as will be discussed later in section RTM3 Thin Sheet Composite Specimens.

Temporary Change of the RH in the Environmental Chamber After Removal of Specimens

We have not examined the duration of the temporary change in RH after a sample was removed from the environmental chamber (the salt bath desiccator). We noticed however, that during the first sorption experiments on powder samples (where the individual samples were removed from the same environmental chamber at a rate of about one sample every 10 minutes), that the initial slopes for the weight changes became flatter with increasing sample number. A repeat of the experiment after 24 hours, with a reversed sequence of sample removal, gave the same result, that is, the first samples taken out of the desiccator showed the highest slope in weight change. This indicates that, as a result of opening the desiccator for sample removal, there is enough change in RH inside of it, to affect the moisture equilibrium concentration of the rapidly sorbing samples remaining inside. This effect is observable for both absorption or desorption experiments.
A similar observation was made even with the slower sorbing thin sheet specimens. Qualitative estimates seem to indicate that one should wait at least for 3 hours before removing the next sample. Or better yet, to use a single, small desiccator per sample.

Rapid Methods for Determining M_∞

As we have pointed out above, the major shortcoming of the thick plate (0.09 inch) method is that it takes from 5 months to a year for the composite sample to become saturated with moisture. In order to speed up the determination of M_∞ we considered two methods:

Method A: Moisture sorption on powder samples, and

Method B: Moisture sorption on very thin (0.010 to 0.016 inch) composite plates.

We compared the sorption data of powder and thin plate samples with results previously obtained from nominally 0.1 inch thick composite plates (see references 4 and 5).

Method A seemed to be the simplest and fastest way to obtain the equilibrium concentration. Resin or composite powders can be readily prepared from the turnings of a 7/32-inch diameter drill bit. Several sources of errors in using powder samples seemed obvious and needed further consideration. The moisture uptake of the dried powder or the desorption of moisture from a sample that was equilibrated at higher than the ambient RH, was expected to be fast enough to change during the weighing process after removal from the desiccator. Indeed, within the time it took to weigh the sample on a magnetically damped semimicro balance (which is about one minute) one could clearly observe a change in weight. Thus, the weighing process may introduce a significant error. Therefore, after removal of the samples from the environmental chamber, it is necessary to extrapolate the weight to the time zero when the sample was removed. Since there is no theory for the sorption curve of a powdered sample, we simply used a linear extrapolation of the change in weight from one to two minutes, back to time zero. The differences in absorption and desorption of these extrapolated weights were then defined as the maximum moisture solubilities of the powder samples, M'_∞, for the corresponding RH values. (We use a prime here to distinguish the result from that of the plate samples). The total time necessary to obtain M'_∞ now takes only two days.

Another source of error is that the surface area of the powder sample is much higher than the surface area of a plate sample. Therefore, the amount of moisture adsorbed on the surface and that absorbed in the bulk of the material, need not be the same. For plate samples, the ratio of surface to bulk is obviously much smaller than for powders and may therefore be neglected. A closer inspection for the validity of using powder samples for this purpose was required. Indeed, it was observed that the weight gains of the powder samples were higher, for both, the vinyl RTM3, as well as for the epoxy composite. The average of the RTM3 powder samples was 27 percent higher than the average of the thick composite laminates. A similar weight gain (in the average 23.5 percent higher than the thin or thick sheet speciemns) was observed in the G10 epoxy composite powder.

Method B requires more time to prepare the samples, because they must be carefully machined to a nominal thickness of 0.010 to 0.016 inches and the time required to achieve equilibrium at a given RH also takes longer than with powder samples (depending on the diffusion coefficient). The drying time can be substantially accelerated in a vacuum oven before the samples are transferred into desiccators with a drying agent such as molecular sieve, drierite or other drying agents. Although the samples gain (or loose) less weight during the weighing process than powder samples do, it is still necessary to extrapolate the weight changes during the weighing process to time zero. Since these samples have a plate geometry, we used a linear regression of the weight gain (or loss) versus the square root of time to extrapolate to time zero. The linear behavior of weight gain versus square route of time is justified from Equation (1) and from Figure 1. For

these extrapolations, weight measurements were taken between 1 and 8 minutes after the samples were removed from the environmental chamber. The differences in the extrapolated absorption and desorption curves back to time zero, after removal from the environmental chambers, were defined as M_∞.

In contrast to the powder samples, the "thin" composite plate samples absorbed, within experimental errors, the same amount of moisture as previously measured thick samples (reference and). Since the relative contributions of the surface areas of the thin and thick samples are small, and about the same, their contribution to the overall solubility is negligible. This clearly indicates that the thin plate approach, Method B, is preferable. However, for a quick, rough estimate, powder samples may be used, after deducting about 25 percent from the ultimate weight gain.

Expected Errors in Diffusion Coefficient as a Result of Errors in Measuring M_∞ or Specimen Thicknesses

One can easily see how an error in measuring the maximum moisture solubility, M_∞, would affect the calculation of the diffusion coefficient. This is especially important if one would use the measured solubility of powder samples, where the surface area contributes to a higher apparent solubility (as we have seen when we measured the solubility of the RTM3 powder, which sorbed 27 percent more moisture than thick plate samples of the same material, that is, M'_∞ is 27 percent higher than M_∞). For assumed values of $D = 1.0$, and $l = 0.5$ (as half the plate thickness), equation 1 gives the calculated values for Mt/M_∞ as are shown in Figure 1 (solid line). As one can see from this figure, if we unknowingly accepted the powder solubility, M'_∞, as the true solubility, and if we had plotted the calculated Mt/M_∞ values from the sorption data of the plate samples, we would have obtained the dotted line, and therefore, a different initial slope. As a result the dotted line would intersect the value $Mt/M'_\infty = 0.5$ at a considerably longer time than expected from equation (4 or 5), and therefore, lead to a lower calculated diffusion coefficient. We can easily estimate the error we would make as follows. If we let the plate sample solubility be 27 percent lower than the powder samples solubility, we would have to take the dotted line for the calculation of the diffusion coefficient. This curve levels out at $Mt/M'_\infty = 0.73$ (where $Mt/M_\infty = 1.0$). Therefore $Mt/M_\infty = 0.5$ is where $MtM'_\infty = 0.356$ ($= 0.5*0.73$). Substituting the corresponding times into equation 4 or 5 we find an error of 46 percent. In Figure 3 we have plotted the expected percent error in the diffusion coefficient versus percent error in solubility measurement (the curve shows only positive values of solubility errors, the negative values are obviously opposite).

Although thin sheet specimens are very useful for a rapid determination of the maximum moisture solubilities in composites, they are less suitable for directly determining the diffusion coefficient. One reason is that the initial slope is very steep, and the time to reach 0.5 for Mt/M_∞ is quite short. Another reason is, that after each sample removal, it takes some time for the desiccator environment, that is, its RH, to recover to its specified value, so that the weighing times become a significant part of the exposure times. This is especially true when the time to reach $Mt/M_\infty = 0.5$ is only several recovery times. Another source for errors in measuring the diffusion coefficient on very thin composite plates is that relative variations in thickness are more pronounced in thin specimens than in thick ones. Also, the thickness measurements of thin specimens becomes less accurate if a

micrometer is used for the measurements. Using equation (5), one can estimate the expected errors in diffusion coefficients resulting from errors in thickness measurements which we have plotted in Figure 4.

One may, however, use thin sheets for a preliminary estimate of the diffusion coefficient corresponding to the laboratory environment, that is, for ambient temperature and ambient RH. In this case the dried sample is removed from the desiccator and left outside (in the balance chamber) for the entire sorption time, while intermittent weighings are performed. If the RH in the laboratory does not change significantly while the sample is kept outside, such estimates may come very close to the actual diffusion coefficient and provide a useful preliminary estimate as we shall show in the result section, Estimate of the Diffusion Coefficient from Thin Laminates. These preliminary diffusion coefficients can be used to calculate desirable specimen thicknesses for more accurate experiments. On the other hand, if there are large RH fluctuations in the laboratory, substantial errors in the estimate can result.

Temporary Change of the RH in the Environmental Chamber after Removal of Specimens

We have not examined the duration of the temporary change in RH after a sample was removed from the environmental chamber (the salt bath desiccator). We noticed however, that during the first sorption experiments on powder samples (where the individual samples were removed from the same environmental chamber at a rate of about one sample every 10 minutes) that the initial slopes for the weight changes became flatter with increasing sample number. A repeat of the experiment after 24 hours, with a reversed sequence of sample removal, gave the same result, that is, the first samples taken out of the desiccator showed the highest slope in weight change. This indicates that, as a result of opening the desiccator for sample removal, there is enough change in RH inside of it, to affect the moisture equilibrium concentration of the rapidly sorbing samples remaining inside. This effect is observable for both absorption or desorption experiments.

RESULTS

In this section we will discuss the results of our investigation that were intended to show that the experimental time for determining diffusion coefficients in composites could be substantially reduced. We have taken the same composite laminate materials from which we had previously measured the maximum moisture sulubilities and diffusion coefficients on thick specimens (as reported in references 4 and 5).

RTM3 COMPOSITE POWDER SAMPLES FROM IMPACTED SANDWICH PANELS

The samples were prepared as described in the Experimental section. Their maximum moisture uptake at 80 percent RH was determined from the difference between

the dry and the 80 percent RH equilibrated sample weights. Because of the rapid moisture gain, these weights had to be obtained by extrapolation to time zero after removal from the environmental chamber. The experimental data for the moisture uptake of dry samples in a 43 percent RH and 23°C environment of the laboratory are shown in Figures 5 through 10.

All weights given in the following figures are, for brevity reasons, only the last two or three significant decimals in units of 10^{-5} grams of the nominally 1 to 1.5 gram sample weights. Only the extrapolated values will be given in full in the Appendix.

Since there is no simple theory of the moisture uptake in powder samples with unknown particle distribution, we have defined a somewhat arbitrary extrapolation procedure for the zero-time weight. We simply extrapolated the sample weights at one and two minutes on the balance in a straight line to time zero. The same procedure was used for the 80 percent RH equilibrated samples that lost weight very rapidly in the 43 percent RH environment of the laboratory. This can be seen in Figures 11 through 16. The data of the individual experimental measurements are given in the Appendix. Table 1 lists the maximum percent weight gain of the individual powder samples and compares them with the same samples that were previously determined on "thick, undamaged" plate samples (equilibrated at 80 percent RH, see reference 5). The powder samples were drilled out of the respective plate specimens. Also listed are the percent differences in weight gain between the powder and plate samples.

Table 1. Comparison of the Maximum Moisture Equilibrium Concentration of RTM3 Thick Plate Composite Samples and their Respective Powder Samples at 80 Percent RH.

Specimen ID	#1	#2	#3	#4	#5	#6	Avg.	Std.Dev.	C. Var.%
Powder Wt%	0.212	0.230	0.207	0.217	0.214	0.221	0.217	0.008	3.675
Plates Wt%	0.167	0.176	0.153	0.199	0.162	0.173	0.172	0.016	9.231
% Difference	27.060	30.499	35.413	9.082	32.579	28.007	27.107	9.340	34.456

The powder samples show a smaller variation than the plate samples, however it is quite obvious that the powder samples show a significantly higher solubility than the respective plate samples. The average is 27 percent higher. We attribute this higher moisture gain in powders to an increased contribution in surface condensation of moisture on the powder.

The conclusion is that the rapid powder method is, at best, only useful for a quick preliminary estimate of the solubility and not for an accurate determination of the moisture diffusion coefficient in composites. The expected error in using the powder method can be estimated from Figure 3.

RTM3 THIN SHEET COMPOSITE SPECIMENS

Specimens from impacted Sandwich Panels

The preparation of the thin sheet composite samples was described in the Experimental section. Four samples were machined from the face sheets of undamaged areas of impacted sandwich panels, as described in reference 5. The weight gain (weight loss) experiments were carried out similarly to the powder samples. The extrapolation procedure for plate samples is well defined by equation (1). We therefore used a least square fit of the sorption data where the weight gains were plotted as the square root of time. The intersection of this line with the ordinate was defined as zero-time weight. The results are given in Figures 17 through 20 for the sorption of the dry samples in an ambient environment of the laboratory (43 percent RH) and in Figures 21 through 24 for the weight loss of 80 percent equilibrated samples in the same ambient environment. The individual experimental data are listed in the Appendix. Table 2 lists the weight gains in thin plate samples and compares them with the average of the plate samples in Table 1.

Table 2. Comparison of the Maximum Moisture Equilibrium Concentration of RTM3 Thin Sheet Composite with Thick Plate Laminates at 23°C and 80 Percent RH.

From Extrapolation	#1	#2	#3	#4	Average	Std.Dev.	C.Var.(%)
80% RH Wt.	1.89642	1.93147	1.92069	1.75597			
dry Wt.	1.89312	1.92810	1.91731	1.75294			
% Wt. gain	0.174	0.175	0.176	0.173	0.175	0.001	0.811
% Wt. gain of thick laminates					0.172	0.016	9.231

Again, we find that the variation in the measured thin sheet specimens was less than that of the thick laminates. The somewhat higher moisture sorption in the thin sheet specimens is well within the error limit of the thick laminates, and is therefore not significant. Because of the small variation in maximum moisture solubility for the thin laminates, we believe that the new value of maximum moisture solubility average of 0.175 (instead of 0.172) is more likely.

The same set of samples were measured after equilibration at 23 percent RH which was maintained in a desiccator above a sodium acetate salt bath (see Figures 25 through 28). The results are listed in Table 3.

Table 3. Maximum Moisture Equilibrium Concentration in RTM3 Thin Sheet Composite Specimens at 23°C and 23 Percent RH.

From Extrapolation	#1	#2	#3	#4	Average	Std.Dev.	C.Var.(%)
23% RH Wt.	1.89396	1.92904	1.91818	1.75373			
dry Wt.	1.89312	1.9281	1.91731	1.75294			
% Wt. gain	0.044	0.049	0.045	0.045	0.046	0.002	4.256

Specimens from an Earlier Fabricated RTM3 Panel (Fab. 1)

We compared the previous results for the RTM3 (Fab. 1) composite that showed a lower maximum moisture solubility than the samples from the undamaged area of the impacted sandwich face sheet laminate (see reference 4). Figures 29 and 30 show the ambient sorption of the dry samples, and Figure 31 and 32 show the moisture loss of the 80 percent RH equilibrated samples in the ambient laboratory environment. In Table 4, we compare the moisture gain in the previously measured RTM3 (Fab. 1) thick laminate (reference 4) with the thin sheet measurements. The experimental data are listed in the Appendix.

Table 4. Comparison of the Maximum Equilibrium Concentration of RTM3 (Fab. 1) Thin Sheet Laminates with the Corresponding Thick Plate Specimens of Reference 4.

From Extrapolation	#1	#2	Average
80% RH Wt.	1.91633	1.92015	
dry Wt.	1.91322	1.91708	
% Wt. gain	0.163	0.160	0.161
% Wt. gain of thick laminates			0.157

The differences between thin sheet and thick laminates of RTM3 (Fab. 1) specimens are within experimental error. Compared with the results of the RTM3 specimens from the undamaged areas of the impacted sandwich panels of Table 2, they show a lower maximum moisture solubility. We find that the difference in equilibrium concentration between the RTM3 from the sandwich panel and that from the previously measured RTM3 (Fab. 1) samples are of the same magnitude in both, the thin and thick specimens.

The maximum equilibrium concentration of these samples were also measured at 23 percent RH. The procedure was the same as described above. The thin samples were equilibrated at 23 percent RH and then the weight gain was measured at ambient laboratory conditions (23°C and 43 percent RH), see Figures 33 and 34. The results are listed in Table 5.

Table 5. Maximum Moisture Equilibrium Concentration in RTM3 (Fab. 1) Thin Composite Sheets at 23 Percent RH.

From Extrapolation	#1	#2	Average
23% RH Wt. dry Wt.	1.91429 1.91322	1.91817 1.91708	
% Wt. gain	0.056	0.057	0.0565

MOISTURE SORPTION IN E-GLASS/G10-EPOXY COMPOSITE POWDER AND THIN SHEET SPECIMENS

We also measured the maximum moisture concentration in E-glass/G10-epoxy composite powder and thin sheet laminates and compared them with the previously measured thick laminates (see reference 4) from the same panel. The sorption and desorption measurements were run as described above.

Figures 35 through 38 show weight gain (of dry) and weigh loss (of 80 RH equilibrated) powder samples in the 43 percent RH laboratory environment. Figures 39 through 42 show the same sorption behavior for G10 thin sheet laminates, and Figures 43 and 44 show the weight gain after equilibration at 23 percent RH. Tables 6 and 7 summarize the results.

Table 6. Comparison of E-Glass/G10-Epoxy Composite Powder, Thin Sheet, and Thick Laminate Sorption Data at 80 Percent RH.

From Extrapolation	Powder #1	Powder #2	Thin Sheet#1	Thin Sheet#2	Thick Sheet
80% RH Wt	1.03148	1.09202	1.56344	1.59244	3.94206
Dry Wt	1.02228	1.08253	1.55191	1.58065	3.91388
% Wt gain	0.900	0.877	0.743	0.746	0.720

Table 7. Maximum Moisture Concentration in E-Glass/G10-Epoxy Thin Sheet Composite Laminate at 23 Percent RH.

From Extrapolation	#1	#2	Average
23% RH Wt dry Wt	1.5563 1.55191	1.58521 1.58065	
%Wt.gain	0.282	0.288	0.0285

As before, we find that the powder samples sorb significantly more moisture than the thin sheet or the thick sheet laminates (on the average 24 percent higher than the thick composite). The thin sheet specimens give, within the experimental error, the same results as the thick sheet.

ESTIMATION OF THE DIFFUSION COEFFICIENT FROM THIN LAMINATES

In the discussion section, we remarked that one should not expect accurate diffusion coefficients from thin composite specimens. However, for ambient conditions (room temperature and the existing RH of the laboratory), one can get a reasonably good estimate of the average diffusion, especially, when the RH value remains within a narrow range of ±2 percent. Such preliminary estimates are quite useful, since they permit one to calculate the sample thicknesses required for more accurate experiments, and, they can be carried out within one day.

The following steps can be used for preliminary estimate of the diffusion coefficients:

1. Determine the maximum equilibrium concentration for the highest RH value of interest (such as 80 percent) and a lower one (between 20 and 35 percent RH). Potassium chloride for 80 percent RH and sodium acetate for 23 percent RH can be used for constant humidity salt solutions.

2. Fit an approximate maximum equilibrium concentration curve as a function of RH through these points and through zero such as shown in Figures 45 and 46.

3. From this curve, estimate the maximum moisture solubility corresponding to the laboratory humidity; this gives M_∞.

4. Remove the dried sample from the desiccator and measure the weight gain of the sample in the laboratory environment.

5. Plot the data as M_t/M_∞ versus (time/thickness^2)$^{1/2}$ in units of seconds and centimeters.

6. Use equation (5) to calculate the diffusion coefficient.

As an example, we show the results of two RTM3 laminates: one sample, (RTM3, #3), from the sandwich panel, and one sample from the RTM3 (Fab. 1), panel (#1). The sample thicknesses (average of nine measured points each) were 0.0403 and 0.0419 cm respectively. The average solubility curves for these materials (obtained from the thin sheet sorption experiments) are shown on Figures 45 and 46 where we have used a quadratic least square fit through the experimental data (four each for the sandwich face materials and two for the Fab. 1 material). In Figure 45 we have overlaid a box indicating what error we would expect if we had a variation in RH from 42 to 62. The error in solubility for this case would be 53 percent which is quite substantial. Thus a monitoring of the RH is necessary. Fortunately, one rarely has such a big change within the measurement times (which is the order of one day, since M_t/M_∞ only has to reach 0.5). The first sample was run when the laboratory RH was 60±1 percent. From the curve in Figure 45, we estimate a maximum moisture solubility in the sample to be 0.126 weight percent. The second sample was run when the laboratory RH was 43±1 percent. From Figure 46 we estimate a solubility of 0.98 weight percent.

Figures 47 through 51 show the thin sheet sorption plots as: weight versus square root of time, M_t/M_∞ versus (time/thickness^2)$^{1/2}$, and the intersection of this curve with $M_t/M_\infty = 0.5$.

The calculated diffusion coefficients for the RTM3 (#3) thin laminate sample was 4.0×10^{-9} cm^2/sec. The corresponding thick sample average diffusion coefficient previously reported was 4.1×10^{-9} cm^2/sec. The RTM3 (Fab. 1, #1) thin sample had a diffusion coefficient of 2.37×10^{-9} cm^2/sec, while the corresponding thick sample diffusion coefficients from the previously measured thick laminates was 2.42×10^{-9} cm^2/sec. From what we have said about possible errors in measuring the diffusion coefficient on thin specimens, such a close fit must be considered fortuitous.

GUIDELINES

From what we have described, we can now present guidelines for more efficient measurements of diffusion in composites. Needless to say, improving measurement efficiency is an important goal. At the same time, data accuracy must be maintained. This is especially true if the resulting data becomes the basis for environmental modeling. In a natural environment, temperature and humidity change continuously. A future report will discuss the dynamics of moisture diffusion. However, we need to generate enough data for a three-dimensional representation of the diffusion coefficient as a function of moisture concentration and temperature used for such models (Equation 9). The first set of guidelines refer to measurements at room temperature. The measurements at elevated temperature are similar, but require more attention to details.

ROOM-TEMPERATURE MEASUREMENTS OF D

The bulk solubility (maximum moisture equilibrium concentration) in a composite is primarily governed by the solubility of moisture in the organic matrix (glass fibers and graphite fibers do not absorb moisture in the bulk of the fiber). However, if there is a lack of interfacial bonding between resin and fiber, or if the void volume fraction is high, the resulting capillary condensation may also contribute significantly to the overall maximum moisture concentration, and result in higher than expected values, especially at high RH values. For room temperature measurements of solubility and diffusion coefficient, we recommend the following steps:

1. Machine thin sheet laminate plate specimens (0.01 to 0.016 inch thickness), measure their thickness along the edges and in the center, and take the average. From these samples determine the bulk solubility of the composite.

2. Dry the specimens in a vacuum oven, at or below temperatures at which the laminate has been cured. This will not take more than a few hours if one can heat the samples above 90°C. Then store the samples in a desiccator over a drying agent such as molecular sieves or dryerite.

3. Determine the moisture sorption at ambient laboratory temperature and humidity conditions over the period of one day. Although, for obtaining the initial dry weight, it usually suffices to stop the measurements after 10 minutes, continuing the measurements to sorb enough moisture to get to $M_t/M_\infty = 0.5$ will permit a rough estimate of the diffusion coefficient at ambient conditions (as indicated below in step 6). Record changes in temperature and RH throughout the measuremnet time. The larger the changes, the less reliable the resulting estimates. Extrapolate the sorbed weights to time zero after removal from the desiccator (as was shown in Figures 17 through 20). This gives the dry sample weight.

4. Place the samples into a desiccator with 80 percent RH environment until they reach equilibrium. This may take 1 or 2 weeks. Again, an extrapolation to zero time is made (as shown in Figure 21 through 24). The moisture solubility corresponding to 80 percent RH is the difference betwee the extrapolation values for the absorption and desorption. During the time necessary for saturating the samples at 80 percent RH, one may also measure powder samples of the composite (as shown in Figures 5 through 16) and deduct 25 percent from the 80 percent equilibrium weight to obtain an approximate solubility as discussed on page 17 (this can be done within 2 days). One will thus obtain a rough estimate of how long the thin sheet samples should remain in the in the 80 percent desiccator before removal for the more accurate solubility measurement.

5. It is advisable to carry out another moisture solubility measurement between 20 and 50 percent RH equilibration. Since most bulk solubility curves between 0 and 80 percent RH have only a small curvature, one may get a reasonably good approximation by using a nonlinear curve fit through these points and through zero. The equilibration time is about the same as that found for 80 percent RH. Again, one determines the equilibrium weight from the zero point extrapolation (as was shown in Figures 25 through 28). Since the equilibration is now closer to ambient RH, one will observe a flatter slope for this extrapolation curve.

6. The moisture equilibrium corresponding to the current laboratory RH is then taken directly from this solubility curve. Now the first crude estimate of the diffusion coefficient can be made from the data of step 3 which are plotted as M_t/M_∞ versus the square root of time/thicknes^2. From the intersection of the initial sorption curve with $M_t/M_\infty = 0.5$, we obtain the diffusion coefficient from equation (5): $D = 0.049/(\text{time/thickness}^2)$ (in units of seconds and centimeters). The diffusion coefficient thus obtained corresponds to the laboratory temperature and humidity.

7. The next step is to calculate sample thicknesses for more accurate measurements. Since we already know the maximum moisture solubilities as a function of RH and the approximate diffusion coefficient, we can now select the specimen's thickness such that $M_t/M_\infty = 0.5$ can be reached within a convenient time (between four to eight days). On page A-7 we have supplied a table where the diffusion coefficients are listed in the first

column. The upper part of the table gives the time (in hours) to reach $M_t/M_\infty = 0.5$ for a given diffusion coefficient and a specified plate thickness. Values in between can be easily obtained by interpolation. The lower part of the table gives the times (in hours) to reach equilibrium saturation for thin sheet laminates. The tables were calculated using equation (1). We have defined the saturation value M_∞ to be 99.3 percent of the true saturation value for practical reasons. First, fluctuations in RH will probably prevent a better accuracy, second, it would take infinitely long to get to equilibrium.

8. Specimens with the desired thickness can now be machined and a more accurate diffusion coefficient can be obtained. The machined samples are dried in a vacuum oven (sufficiently long to ensure that they are dry). If necessary, one can estimate the drying time from the approximate diffusion coefficient and an assumed activation energy of 12 Kcal/Mole). A few days will suffice for most cases.

9. Now, one can determine the diffusion coefficient for any desired concentration by exposing the sample to an RH that corresponds to the specified concentration. This is usually accomplished by storing the samples above specific, saturated salt bath containers. Many salt-water mixtures, where there is still excess, undissolved salt in the mixture, will produce a constant RH value, depending on the specific kind of salt. Some of these salt solutions change very little with temperature.[8] Since one already knows the approximate time it will take to reach $M_t/M_\infty = 0.5$; it will suffice to make only one or two weight determinations and thus reduce the disturbance of the environment to a minimum. We have said, the sorption curve, plotted as M_t/M_∞ versus square root of time/thickness^2 is a straight line in the range between zero and 0.5. Therefore, being close to 0.5 is good enough to make an extrapolation to 0.5 in order to calculate the diffusion coefficient using equation (5).

ELEVATED TEMPERATURE MEASUREMENTS OF D

The determination of diffusion coefficients at higher than room temperature is not substantially different. One obvious difference is that one cannot use very thin composite plates to obtain the maximum moisture solubility at these elevated temperatures. During the process of removing a thin sample from the heated environmental chamber, cooling it down to room temperature, and weighing it, would lead to an uncontrollable weight loss (or gain) and therefore result in a considerable error, even if one tries to extrapolate back to zero time. Thus, the major concern is to reduce the experimental errors caused by this process. The removal from the environmental chamber and the cool-down to room temperature must be done as quickly as possible. How quick is quick enough? And how long do we have to leave the sample in the elevated temperature chamber to get it to its maximum equilibrium concentration with the RH in the chamber?

Having done the room-temperature measurements first, we are not totally ignorant and may proceed as follows:

1. Estimate the diffusion coefficient at the specific elevated temperature (such as 35 or 55°C). From the room temperature diffusion coefficient, and from the assumption that the temperature coefficient of the diffusion is in the order of 12 Kcal/Mole, we can use

equation (7 or 8) to get a rough estimate of what the diffusion coefficient is at 35 or 55°C (see also Figure 2).

2. With the help of the table on A-7 we can estimate a sample thickness that will get to equilibrium in about two weeks. Such samples are machined and dried. The dry weight is determined and the sample is then placed into the preheated temperature chamber at the specified RH.

3. Since the rate of diffusion changes exponentially with temperature, a rapid quenching from the oven temperature to room temperature is required. Therefore, have two metal blocks ready that are kept at room temperature or slightly below room temperature and place the sample between them as soon as they are removed from the chamber. The sample will essentially be at room temperature in 20 to 30 seconds. Now the weight can be determined (if necessary by zero time extrapolation, where zero time is again the time when the sample is removed from the environmental chamber). This yields M_∞ for the elevated temperature and the RH of the chamber. The sample should be replaced into the elevated temperature chamber and re-measured the next day to ascertain that equilibrium has been reached.

4. Now we can determine the diffusion coefficient. The sample is re-dried and place again into the temperature chamber. This time, it is left in the chamber until M_t/M_∞ is about 0.5. From the time of the intersection of $M_t/M_\infty = 0.5$ with the sorption curve (square root of time/thickness^2) the diffusion coefficient is calculated as we did for the room temperature experiments. Every sample removal requires a readjustment of temperature and humidity in the chamber which are not precisely known or controllable. Consequently, there are fewer sources of error when the sample is removed only for the final weighing. If the exposure time to reach a value for $M_t/M_\infty = 0.5$ is inconveniently short or if the measured value exceeds 0.6 when the sample is removed, it is advisable to repeat step 4 with a thicker specimen. Thicker specimens are less sensitive to these errors because the relative amount that is absorbed or desorption is less, and the time to do it is inherently longer.

For environmental modeling, one should know the diffusion coefficients at least for two (better three) reasonably spaced temperatures and humidity concentrations.

CONCLUSIONS

In most organic polymers and composites, moisture diffusion is very slow. It is many orders of magnitude slower than thermal diffusivity. This permits one to easily separate thermal and molecular transport phenomena. It also makes moisture diffusion measurements a very time consuming process. To increase the efficiency of determining the moisture diffusion in composites, we investigated an iterative process, where the measurements are carried out on a set of specimens rather than on single plate specimens.

The most time consuming measurement is the determination of the maximum moisture concentration sorbed in equilibrium with the surrounding RH of the environment. Composite powders and very thin sheets samples of composites were investigated as samples for reducing the sorption times. While powder samples reached the equilibrium concentration very quickly, their surface contribution to the sorption gave about 25 percent higher solubility values than those measured in plate specimens of composite laminates. Although thin sheet laminates took longer to equilibrate than powders, they are sufficiently fast for this purpose, and what is more important, they gave essentially the same results as thick laminates.

Thus an iterative process was proposed to accelerate moisture diffusion measurements substantially without sacrificing accuracy. Different specimen thicknesses are recommended for obtaining the equilibrium solubilities (M_∞), for determining the initial slopes of M_t/M_∞, and for elevated temperature measurements.

This approach requires that more test samples be machined, but the experimentation time can be reduced from many months to a view weeks.

RECOMMENDATIONS

The next step in combining the moisture diffusion behavior in composites with the environment is, to describe the environment such that it will lead to manageable boundary conditions for the diffusion equation to be solved for ship composite structures in various marine environments. The mathematical model for accomplishing this goal has been established. What needs to be done is, to obtain temperature and humidity data from various ocean environments, such as Persian Gulf, Atlantic, tropical and arctic waters, and to use kinetic averaging of the diffusion, so that the variable boundary conditions of the natural environment can be converted into simple, constant boundary conditions.
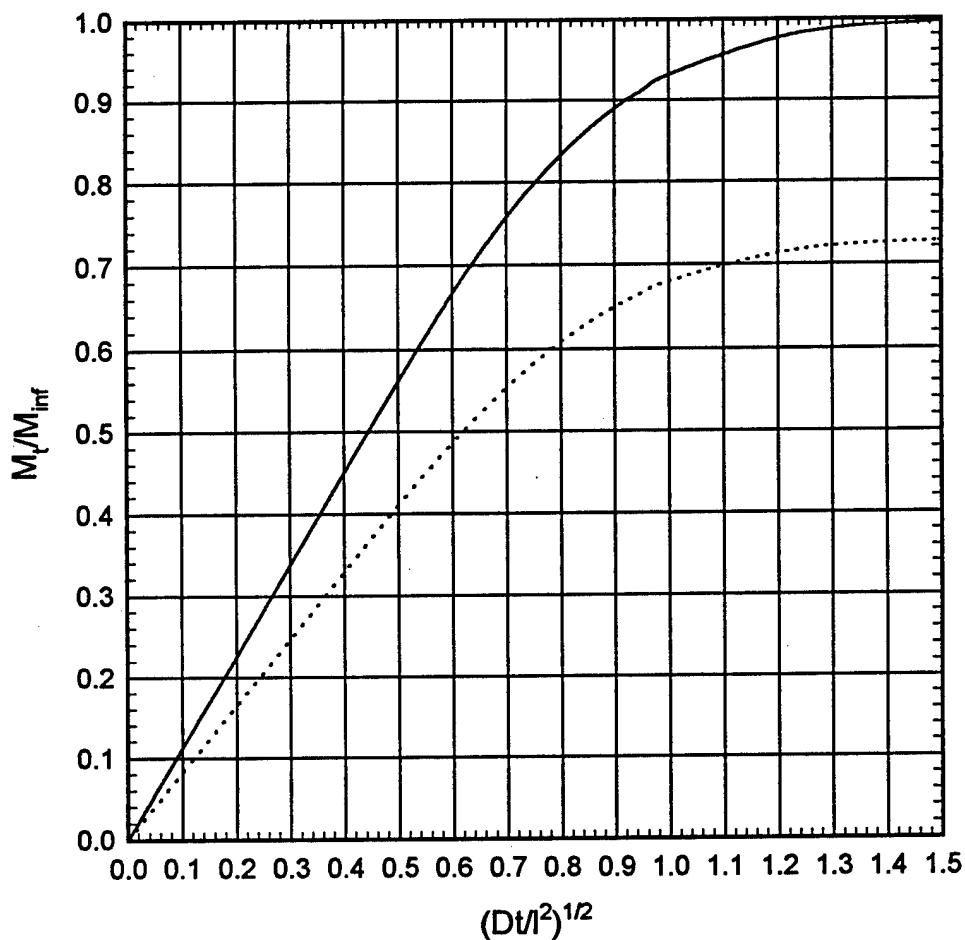
Figure 1. Dimensionless Sorption Plot of Equation (1) (Solid Line) and 27 Percent Reduced Values (Dotted Line).
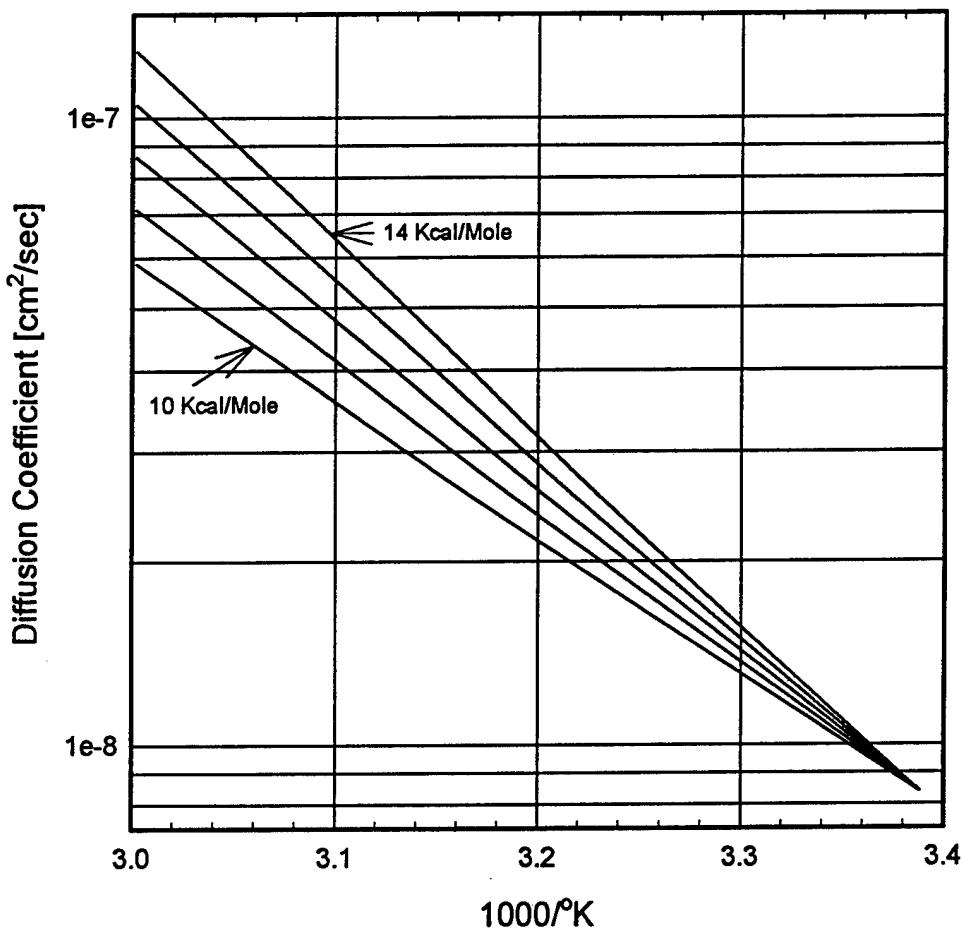
Figure 2. Calculated Change in Diffusion Coefficient for Activation Energies from 10 to 14 Kcal/Mole as a Function of Temperature (°K) for a Given Diffusion Coefficient at 22°C.
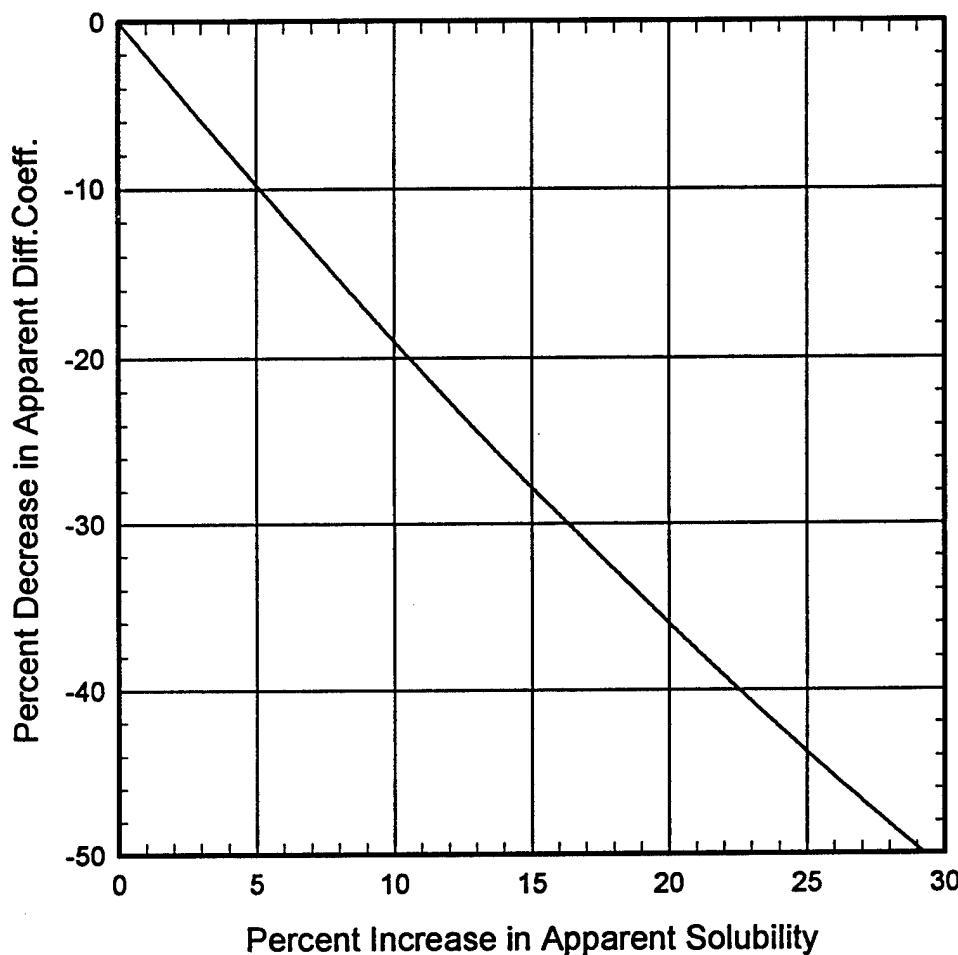
Figure 3. Expected Error in Diffusion Coefficient as a Function of the Measurement Error in Maximum Moisture Solubility for a Given RH Value.
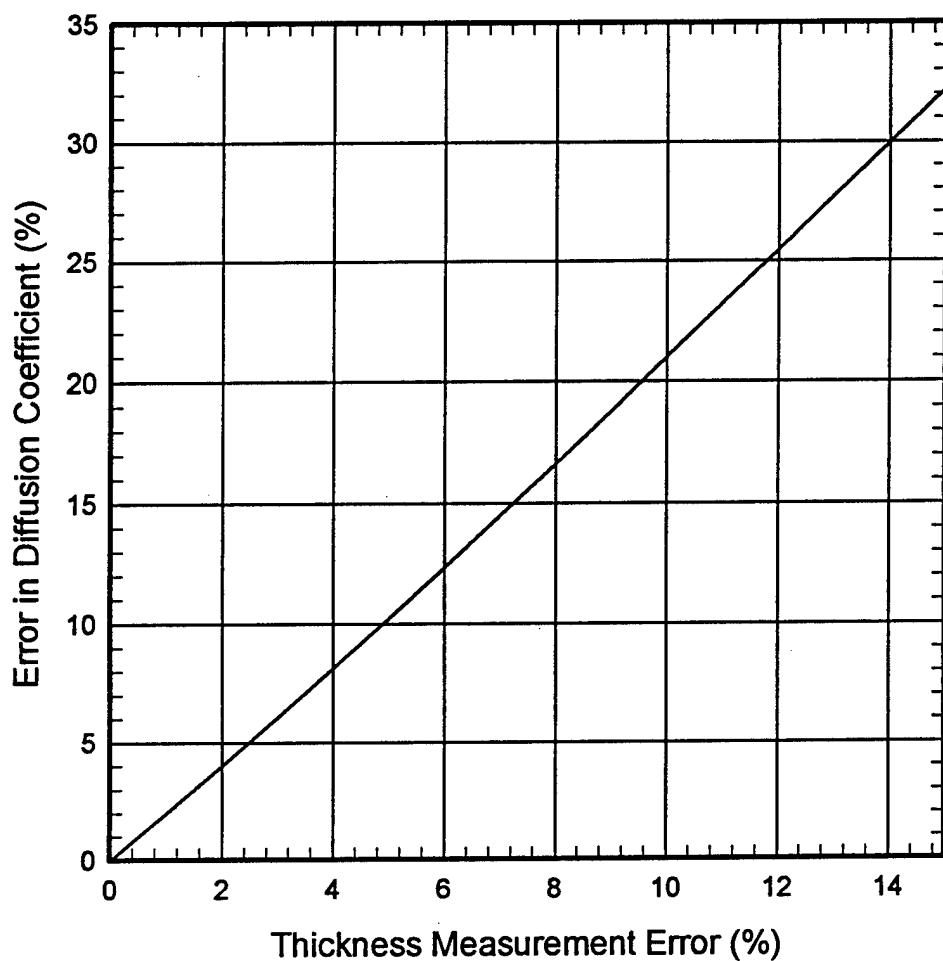
Figure 4. Expected Error in Diffusion Coefficient Resulting from Error in Thickness Measurements.
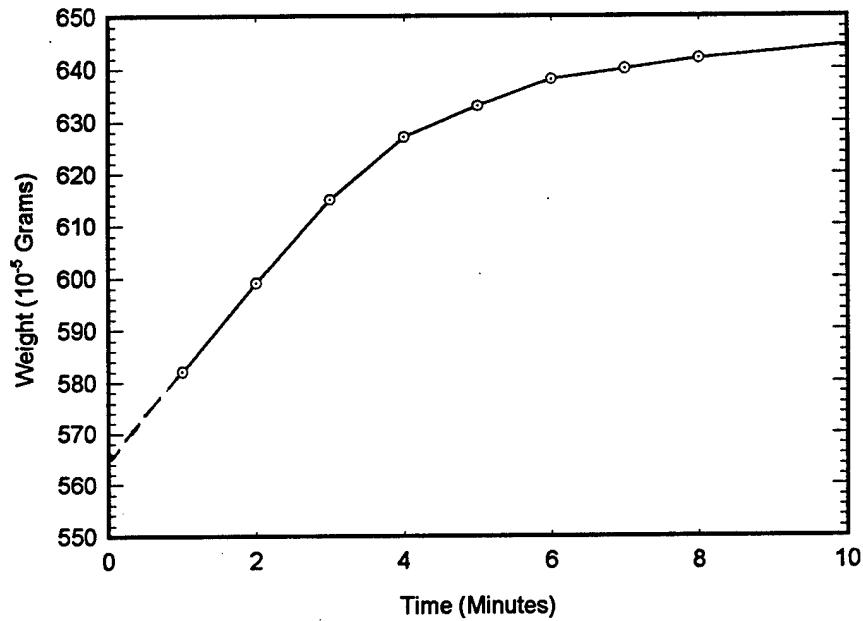
Figure 5. Moisture Gain in Dried RTM3 Composite Powder at 23°C and 43 Percent RH (Sample 1).
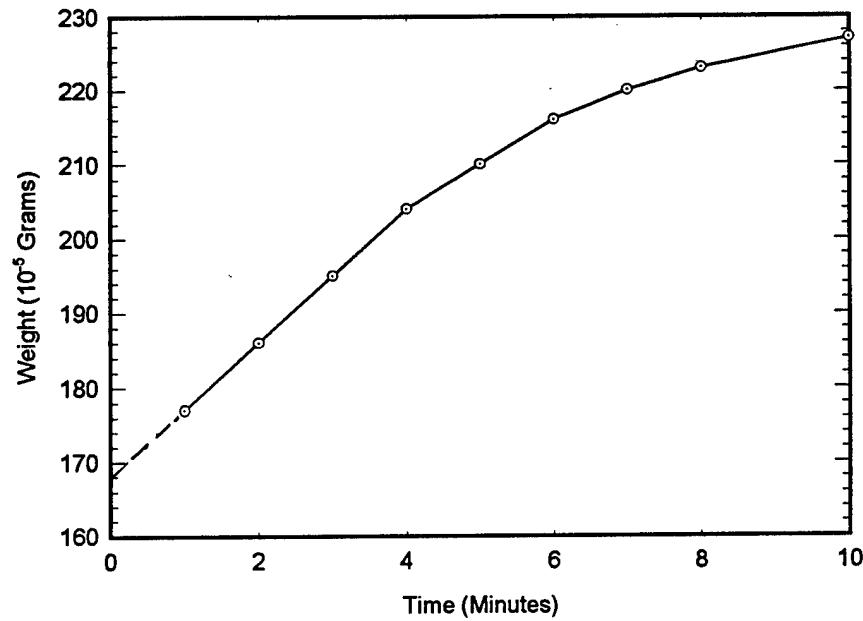
Figure 6. Moisture Gain in Dried RTM3 Composite Powder at 23°C and 43 Percent RH (Sample 2).
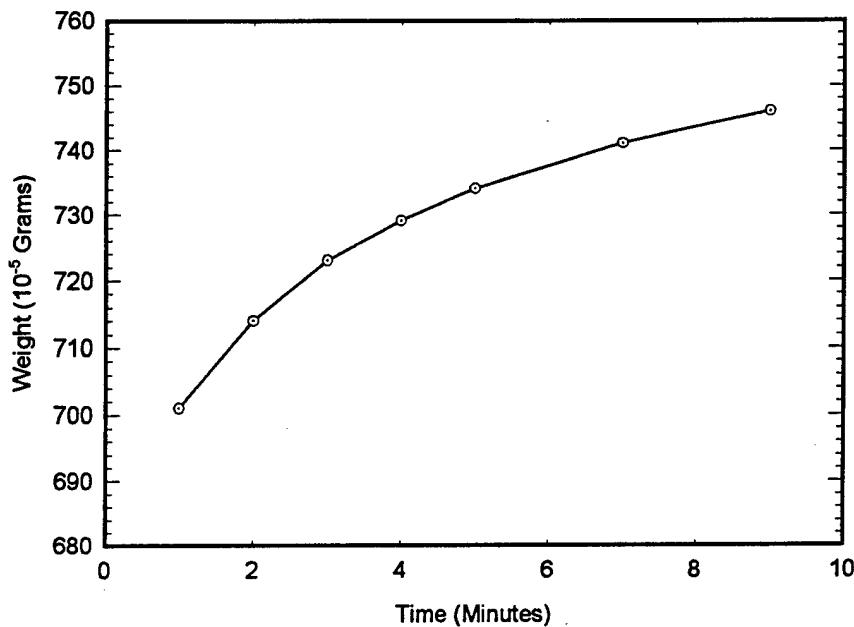
Figure 7. Moisture Gain in Dried RTM3 Composite Powder at 23°C and 43 Percent RH (Sample 3).
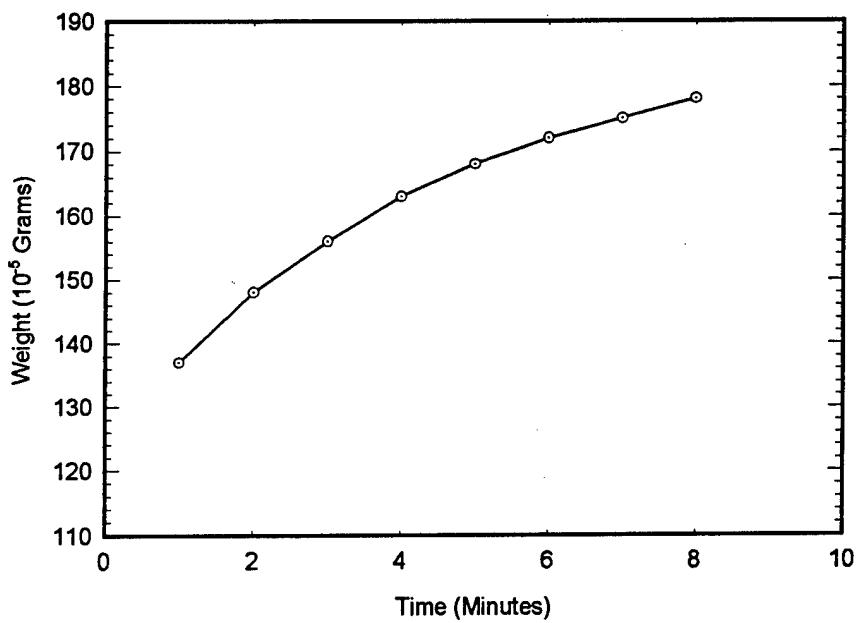
Figure 8. Moisture Gain in Dried RTM3 Composite Powder at 23°C and 43 Percent RH (Sample 4).
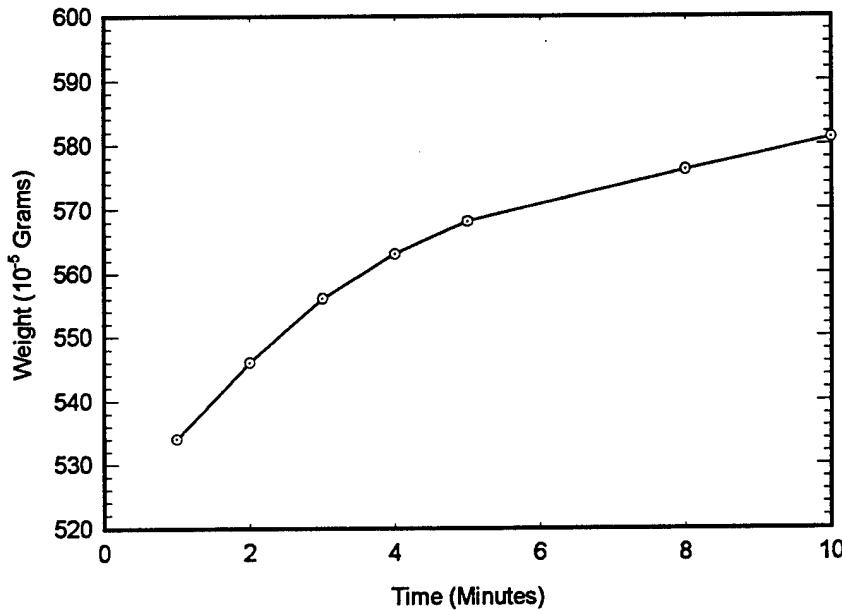
Figure 9. Moisture Gain in Dried RTM3 Composite Powder at 23°C and 43 Percent RH (Sample 5).
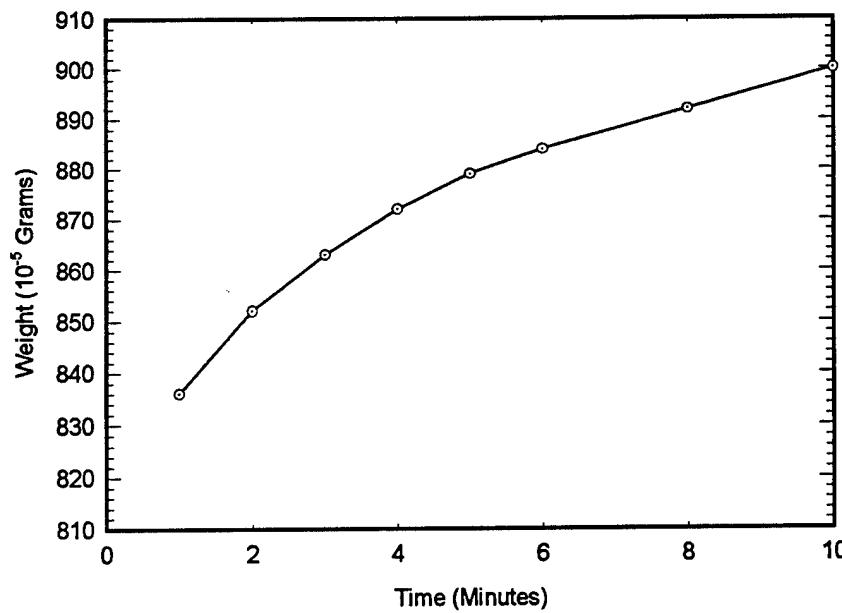
Figure 10. Moisture Gain in Dried RTM3 Composite Powder at 23°C and 43 Percent RH (Sample 6).
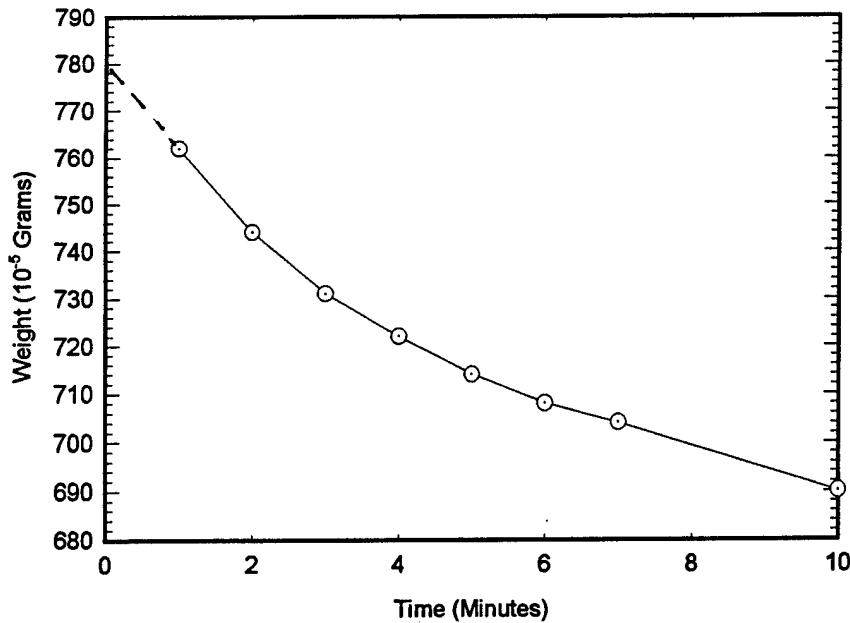
Figure 11. Moisture Loss in 80 Percent RH Equilibrated RTM3 Composite
Powder at 23°C and 43 Percent RH (Sample 1).
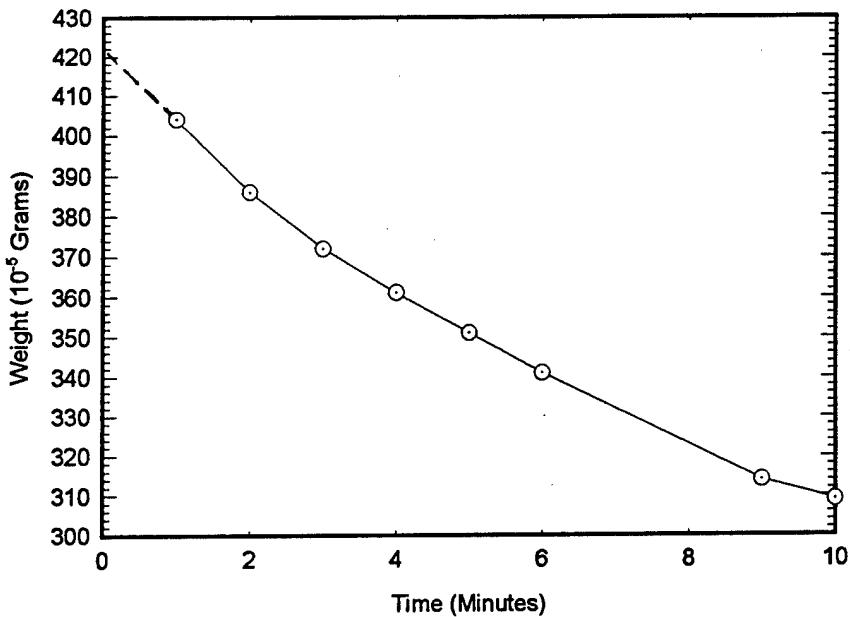
Figure 12. Moisture Loss in 80 Percent RH Equilibrated RTM3 Composite
Powder at 23°C and 43 Percent RH (Sample 2).
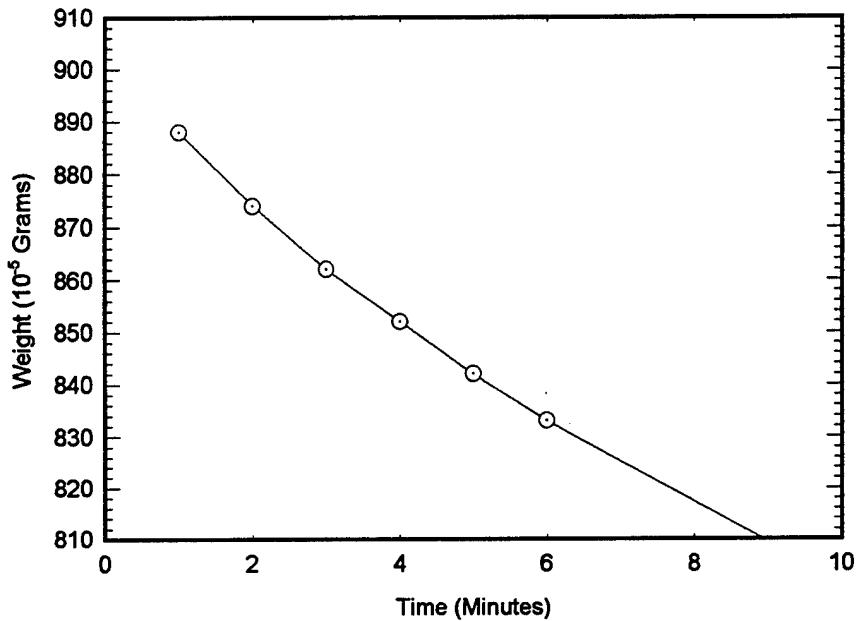
Figure 13. Moisture Loss in 80 Percent RH Equilibrated RTM3 Composite Powder at 23°C and 43 Percent RH (Sample 3).
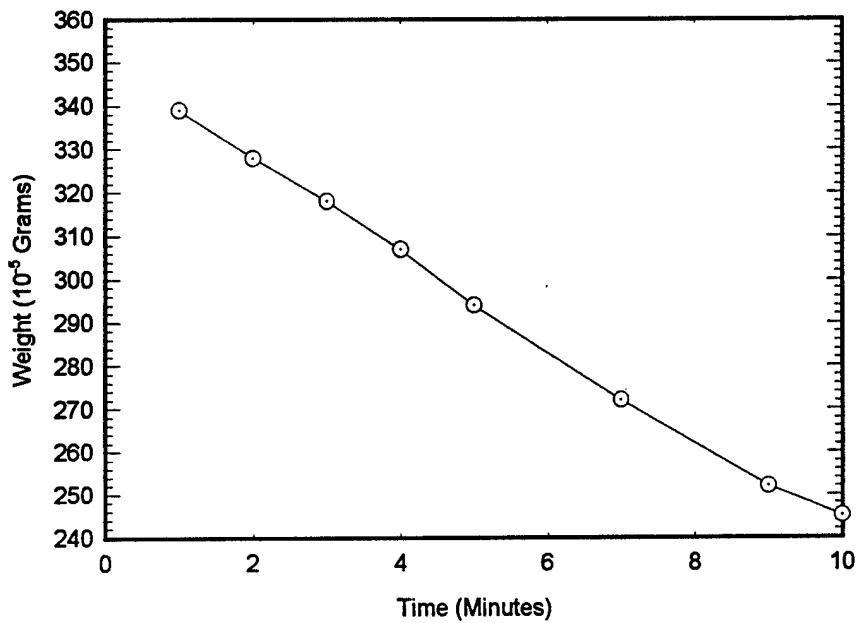
Figure 14. Moisture Loss in 80 Percent RH Equilibrated RTM3 Composite Powder at 23°C and 43 Percent RH (Sample 4).

31
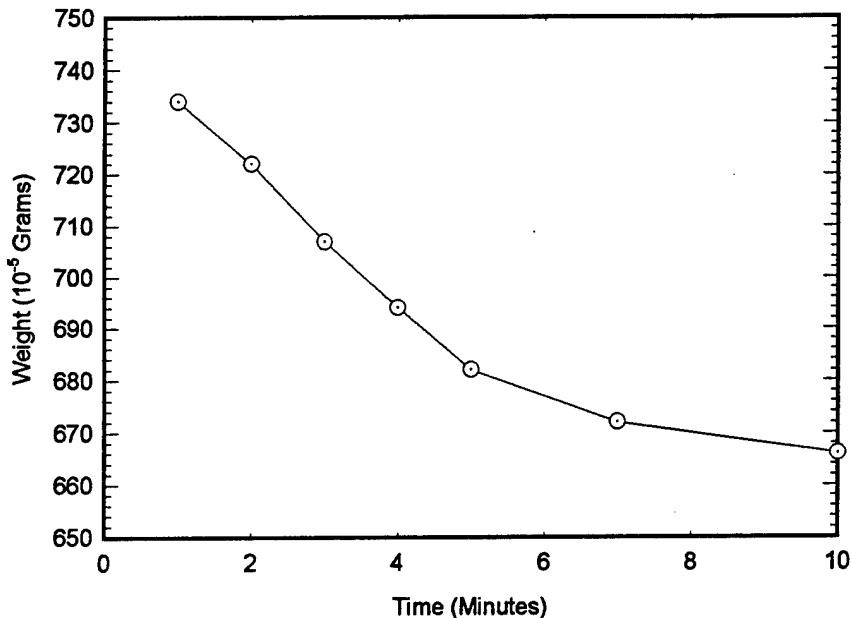
Figure 15. Moisture Loss in 80 Percent RH Equilibrated TRM3 Composite Powder at 23°C and 43 Percent RH (Sample 5).
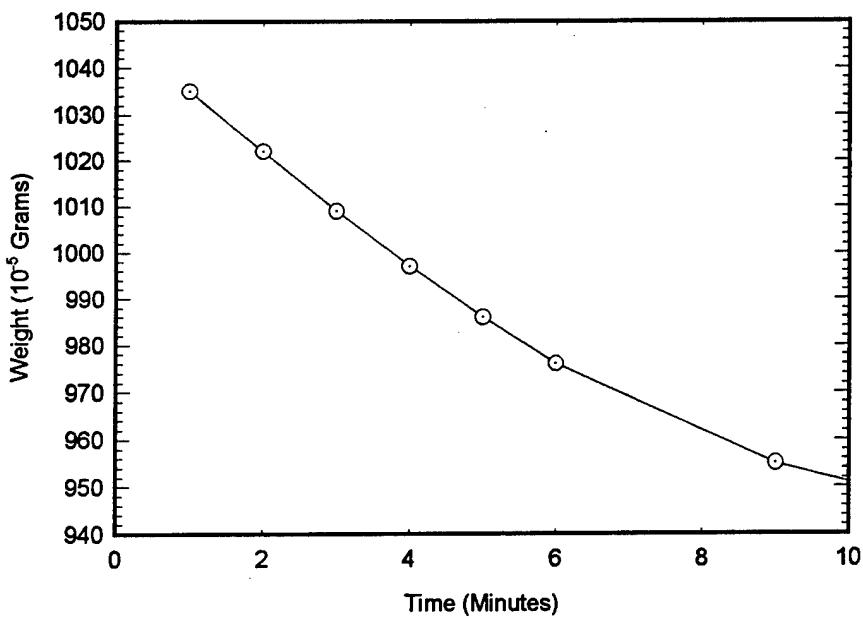
Figure 16. Moisture Loss in 80 Percent RH Equilibrated RTM3 Composite Powder at 23°C and 43 Percent RH (Sample 6).
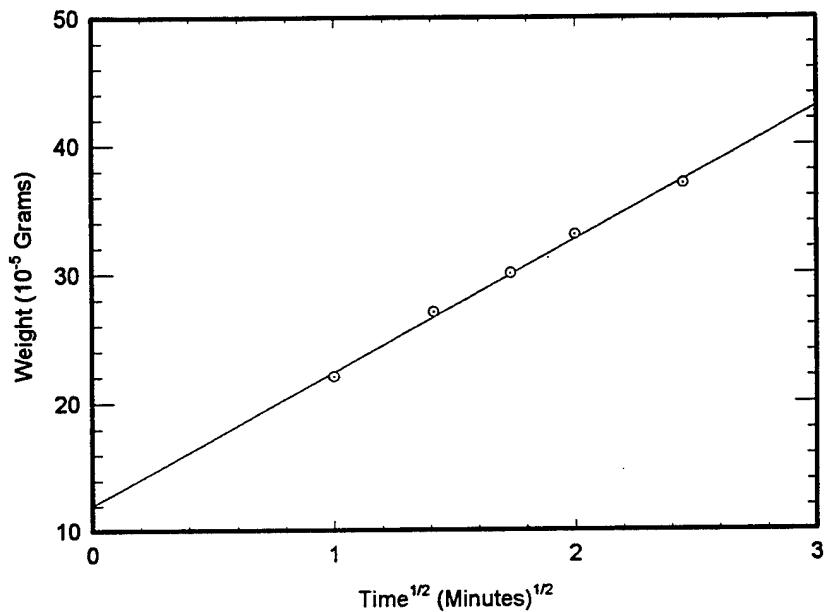
Figure 17. Moisture Gain in Dried Thin Sheet RTM3 Composite at 23°C and 43 Percent RH (Sample 1).
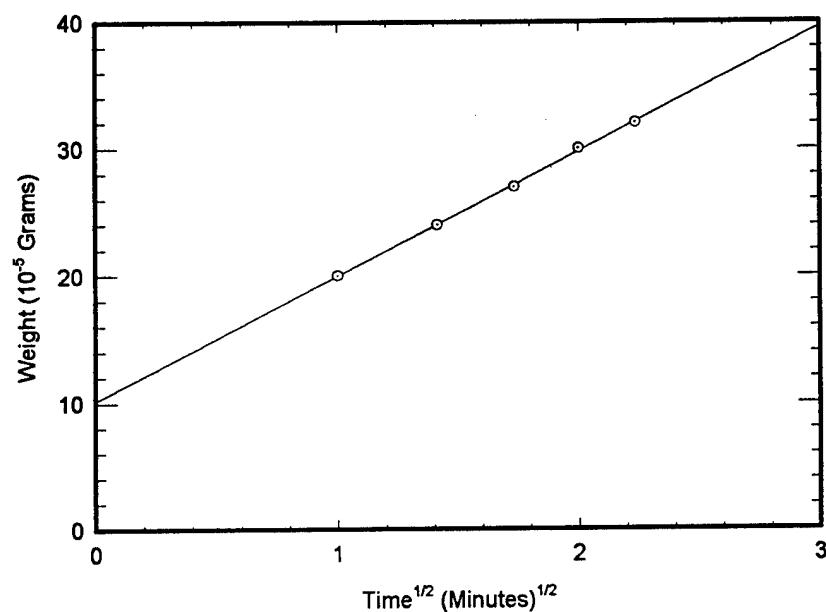
Figure 18. Moisture Gain in Dried Thin Sheet RTM3 Composite at 23°C and 43 Percent RH (Sample 2).
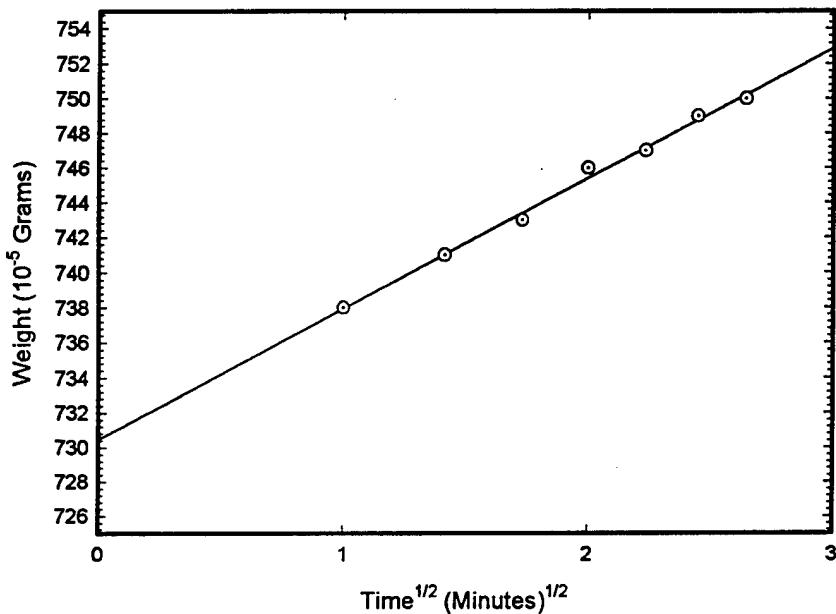
Figure 19. Moisture Gain in Dried Thin Sheet RTM3 Composite at 23°C and 43 Percent RH (Sample 3).
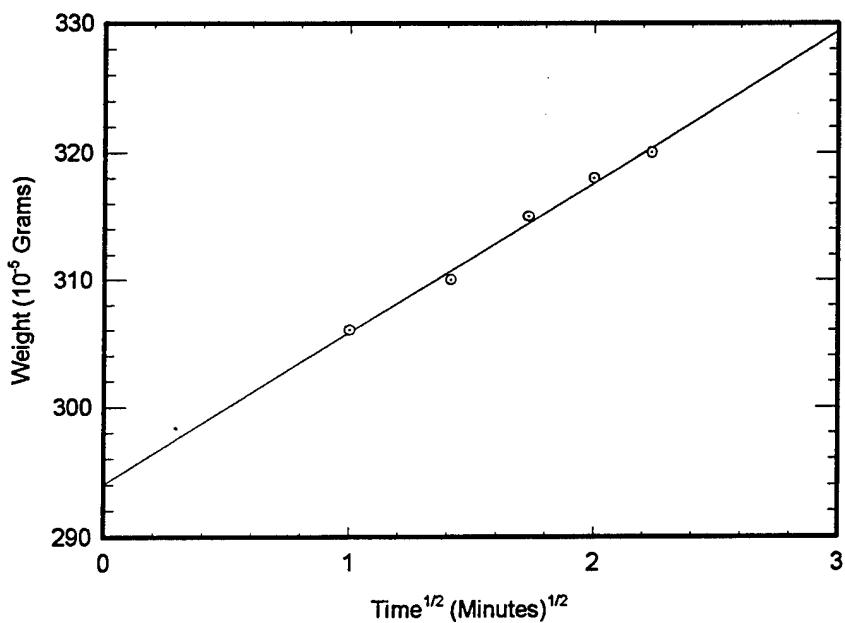
Figure 20. Moisture Gain in Dried Thin Sheet RTM3 Composite at 23°C and 43 Percent RH (Sample 4).
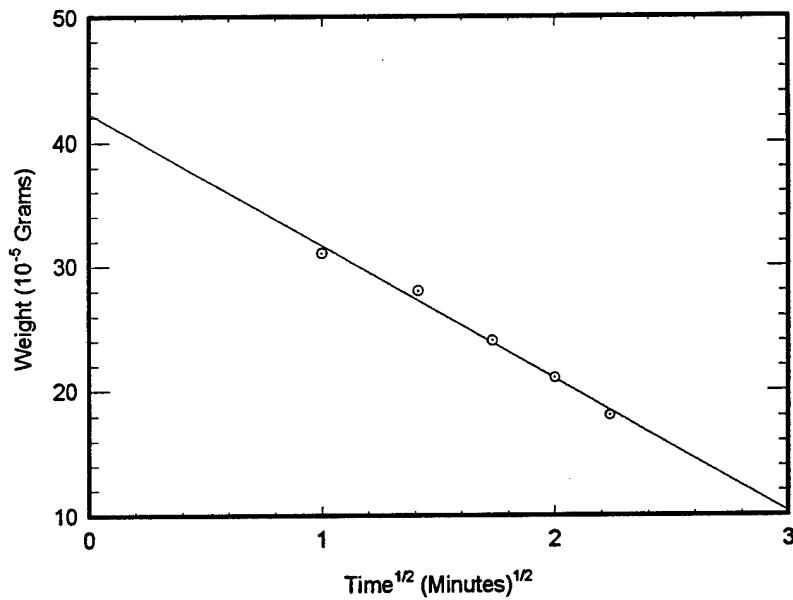
Figure 21. Moisture Loss in 80 Percent RH Equilibrated Thin Sheet RTM3 Composite at 23°C and 43 Percent RH (Sample 1).
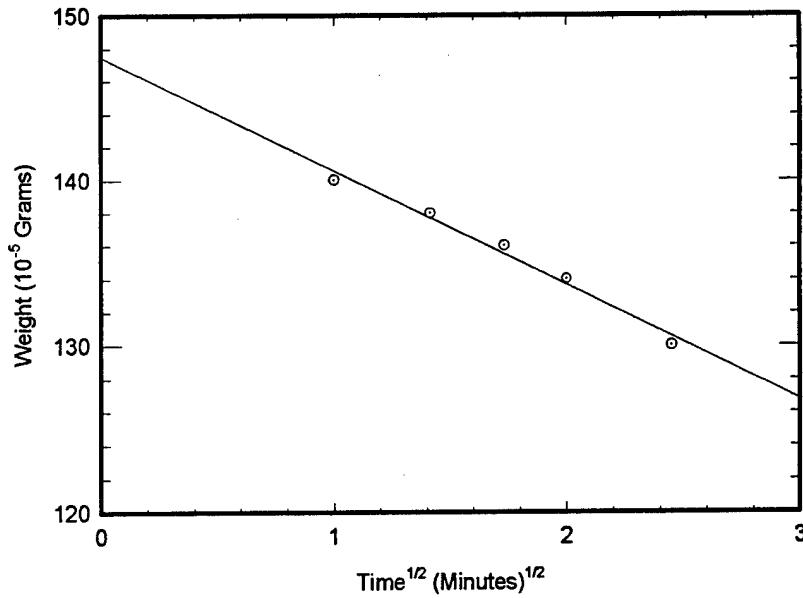
Figure 22. Moisture Loss in 80 Percent RH Equilibrated Thin Sheet RTM3 Composite at 23°C and 43 Percent RH (Sample 2).
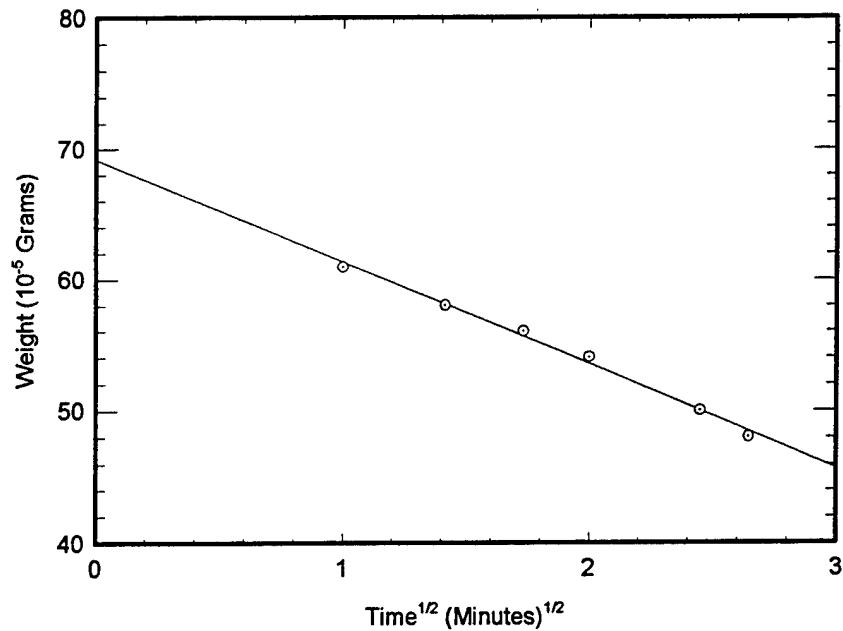
Figure 23. Moisture Loss in 80 Percent RH Equilibrated Thin Sheet RTM3 Composite at 23°C and 43 Percent RH (Sample 3).
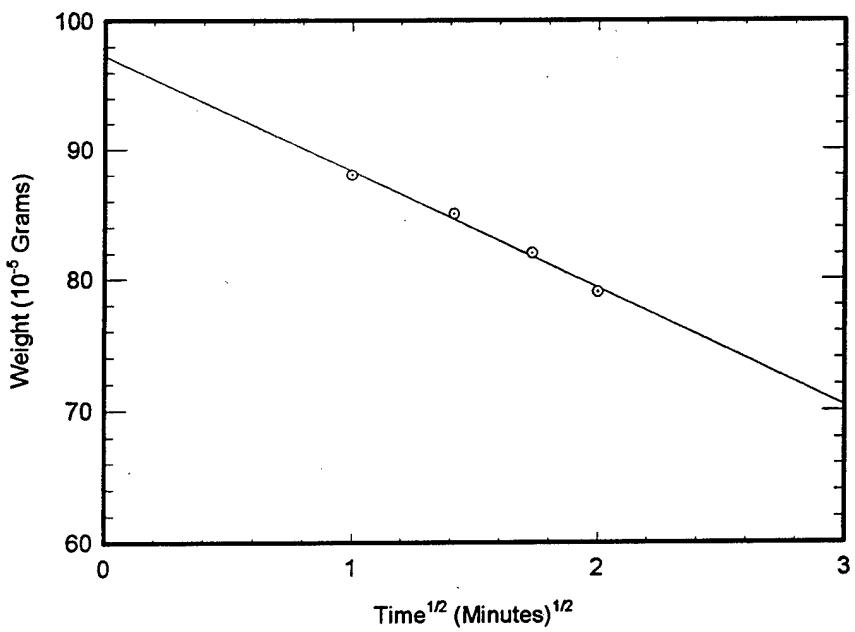
Figure 24. Moisture Loss in 80 Percent RH Equilibrated Thin Sheet RTM3 Composite at 23°C and 43 Percent RH (Sample 4).
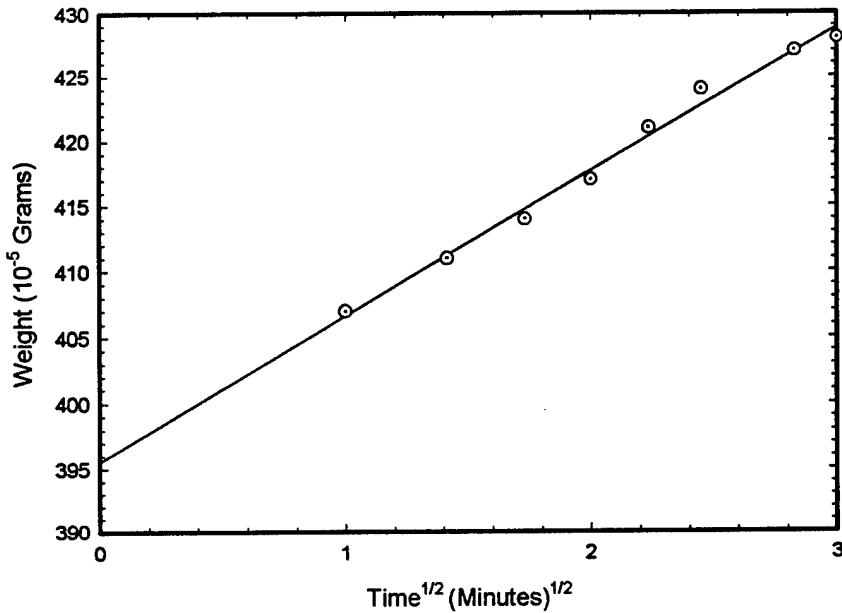
Figure 25. Moisture Gain in 23 Percent RH Equilibrated Thin Sheet RTM3 Composite at 23°C and 43 Percent RH (Sample 1).
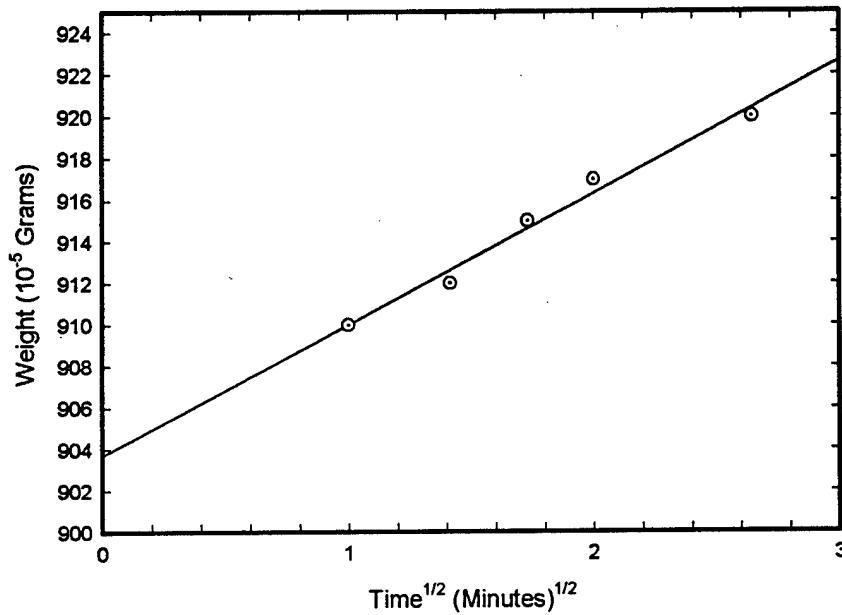
Figure 26. Moisture Gain in 23 Percent RH Equilibrated Thin Sheet RTM3 Composite at 23°C and 43 RH (Sample 2).
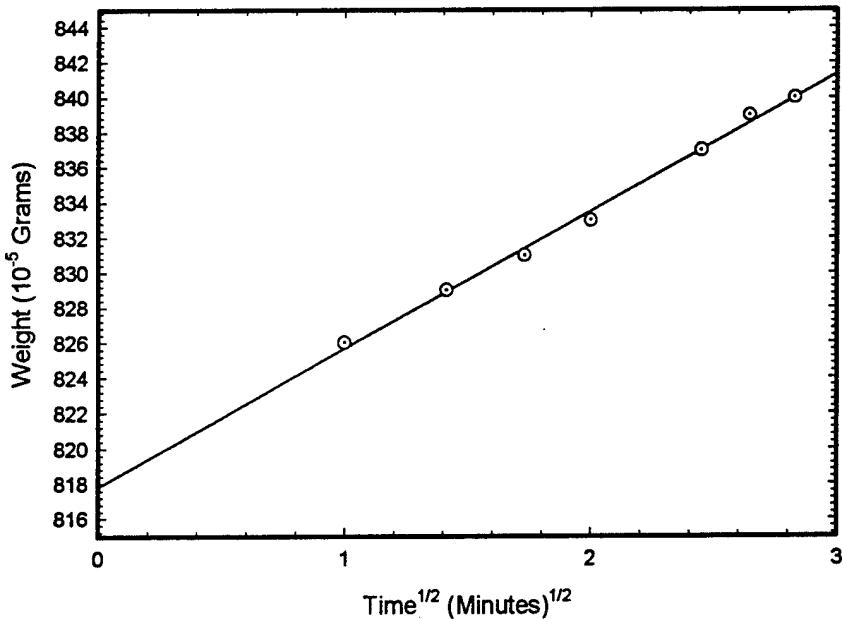
Figure 27. Moisture Gain in 23 Percent RH Equilibrated Thin Sheet
RTM3 Composite at 23°C and 43 Percent RH (Sample 3).
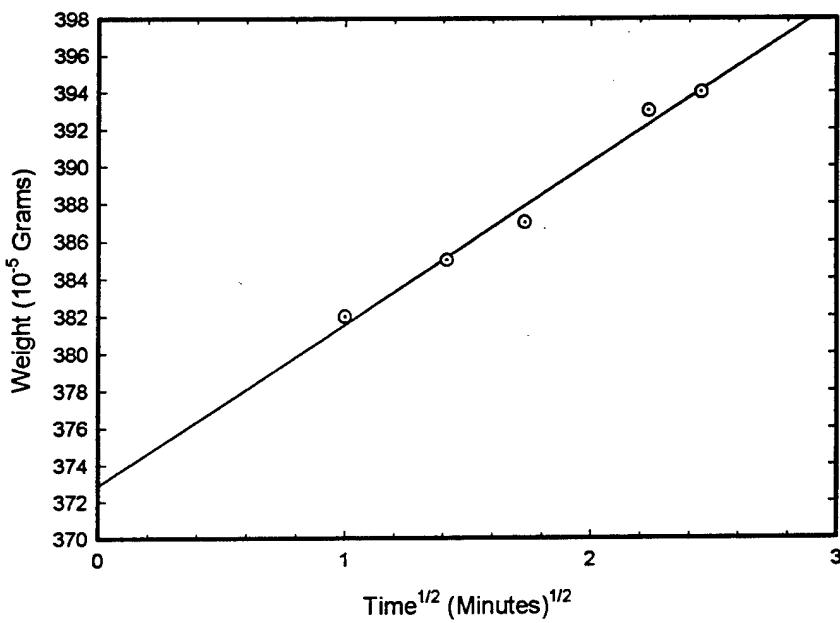
Figure 28. Moisture Gain in 23 Percent RH Equilibrated Thin Sheet
RTM3 Composite at 23°C and 43 RH (Sample 4).
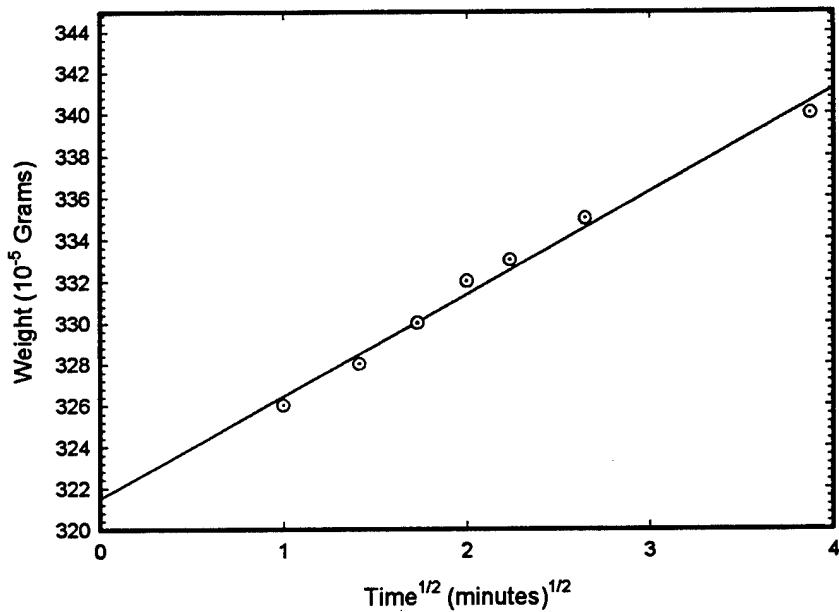
Figure 29. Moisture Gain in Dried RTM3 (Fab. 1) Thin Sheet Composite at 23°C and 43 Percent RH (Sample 1).
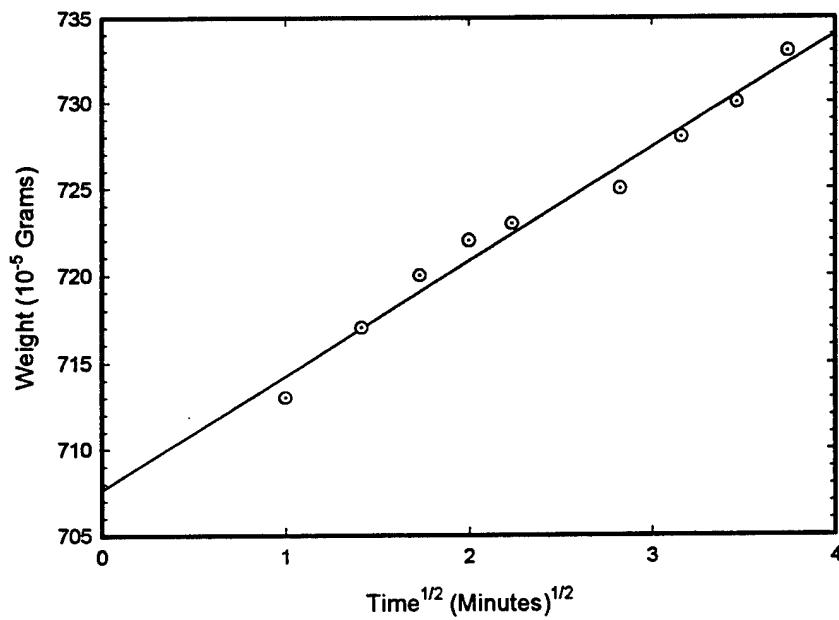
Figure 30. Moisture Gain in Dried RTM3 (Fab. 1) Thin Sheet Composite at 23°C and 43 Percent RH (Sample 2).
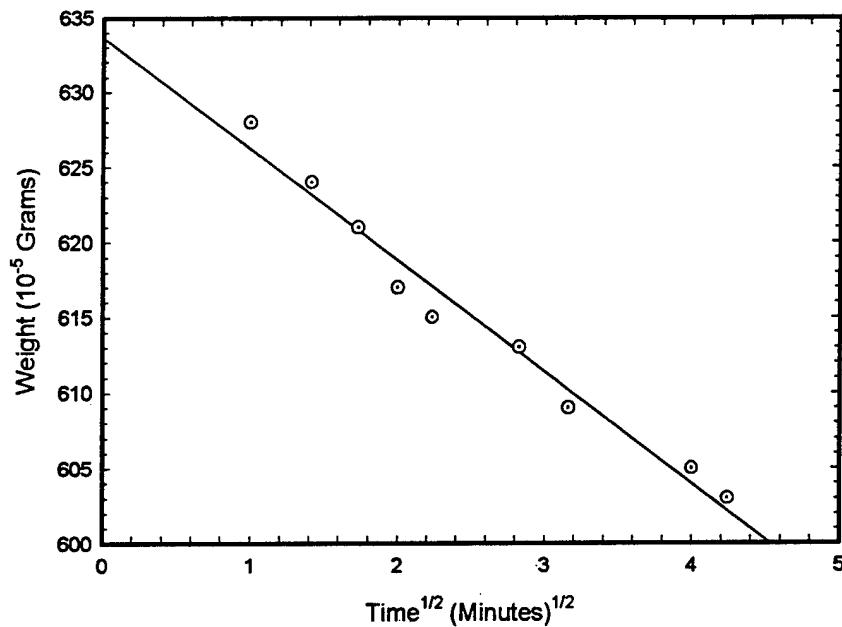
Figure 31. Moisture Loss in 80 RH Percent Equilibrated Thin Sheet RTM3 (Fab. 1) Composite at 23^0C and 43 RH (Sample 1).
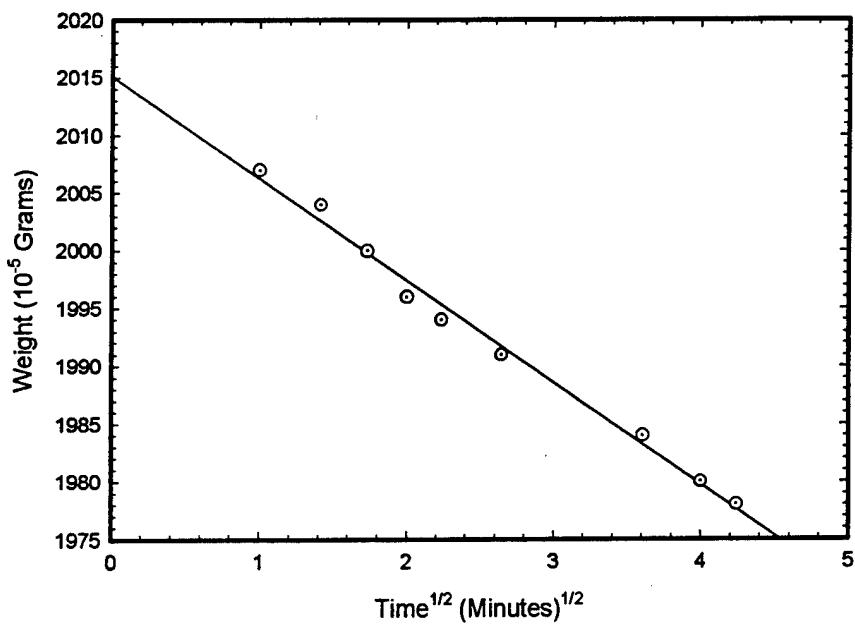
Figure 32. Moisture Loss in 80 Percent RH Equilibrated Thin Sheet RTM3 (Fab. 1) Composite at 23^0C and 43 RH (Sample 2).
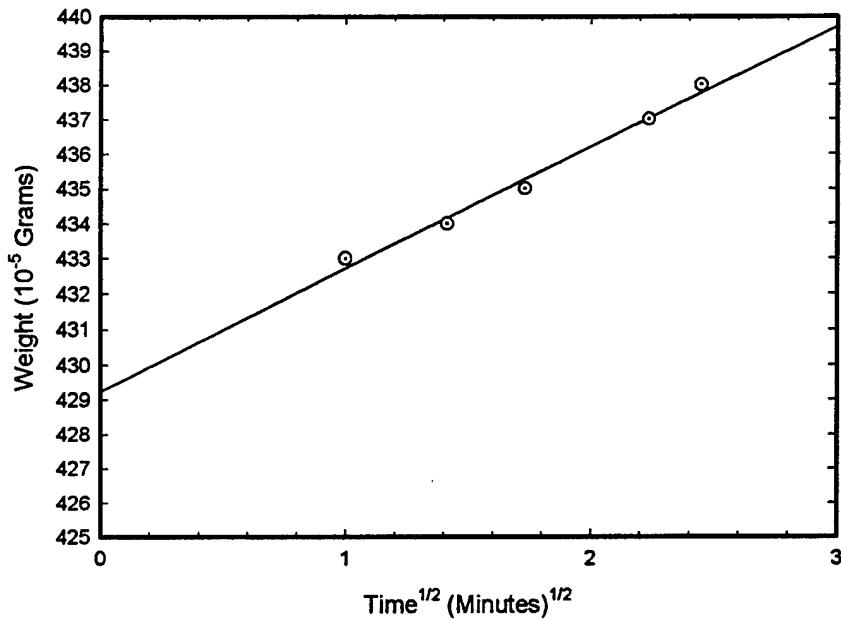
Figure 33. Moisture Gain in 23 Percent RH Equilibrated Thin Sheet
RTM3 (Fab. 1) Composite at 23°C and 43 Percent RH (Sample 1).
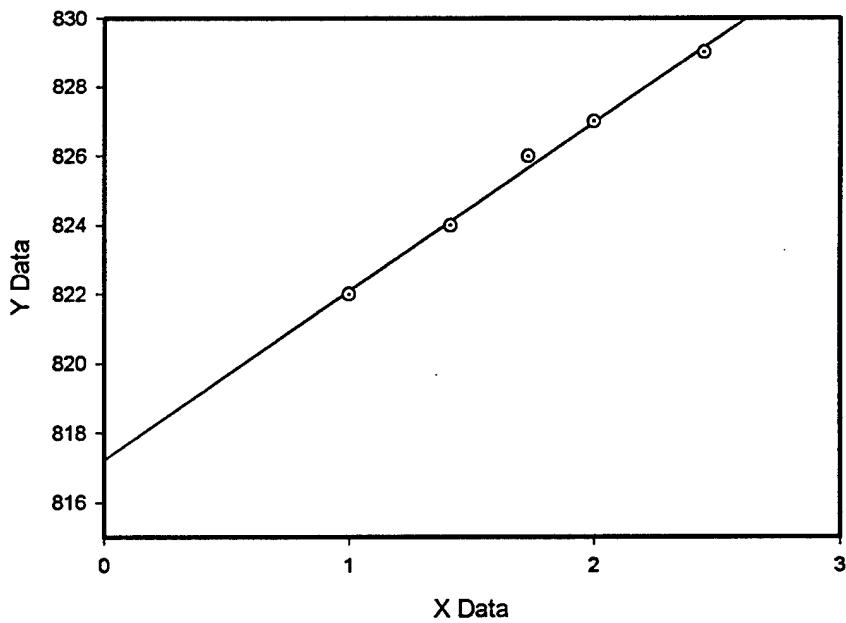
Figure 34. Moisture Gain in 23 Percent RH Equilibrated Thin Sheet
RTM3 (Fab. 1) Composite at 23°C and 43 Percent RH (Sample 2).
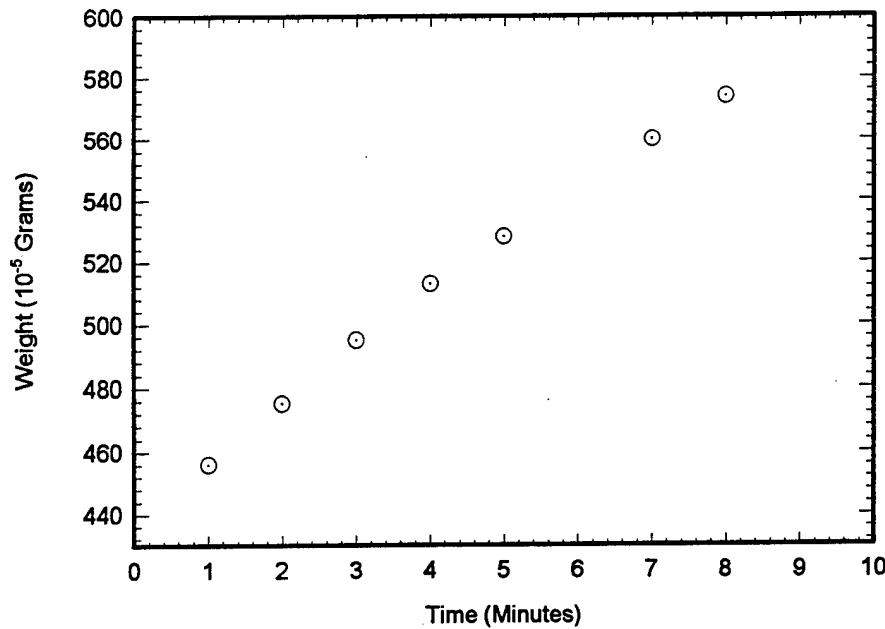
Figure 35. Moisture Gain in Dried G10 Composite Powder at 23°C and 43 Percent RH (Sample 1).
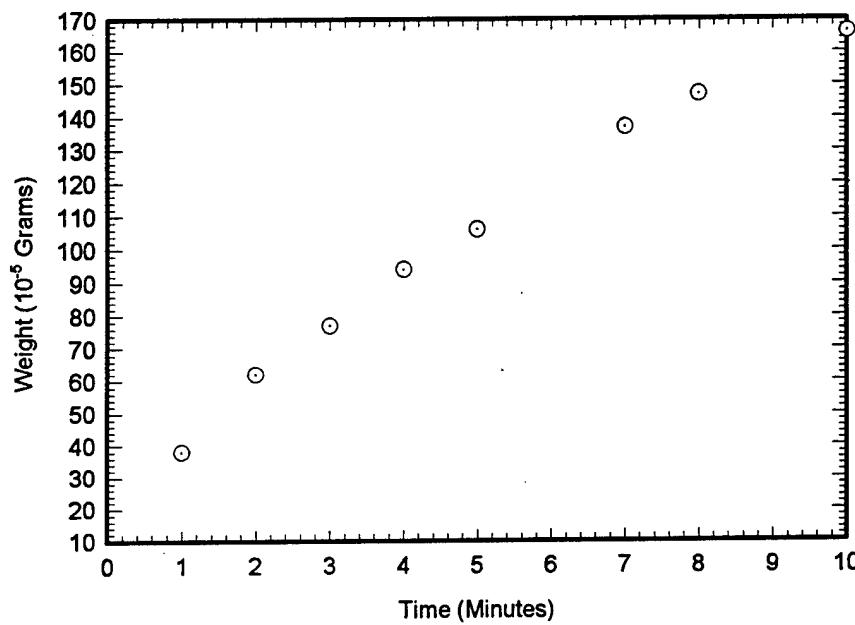
Figure 36. Moisture Gain in Dried G10 Composite Powder at 23°C and 43 Percent RH (Sample 2).
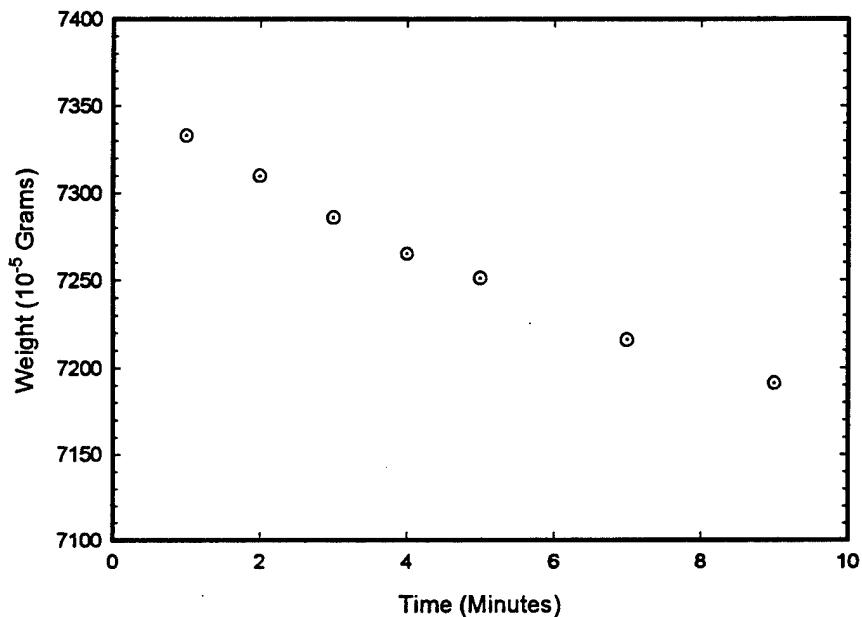
Figure 37. Moisture Loss in 80 Percent RH Equilibrated G10 Composite Powder at 23°C and 43 Percent RH (Sample 1).
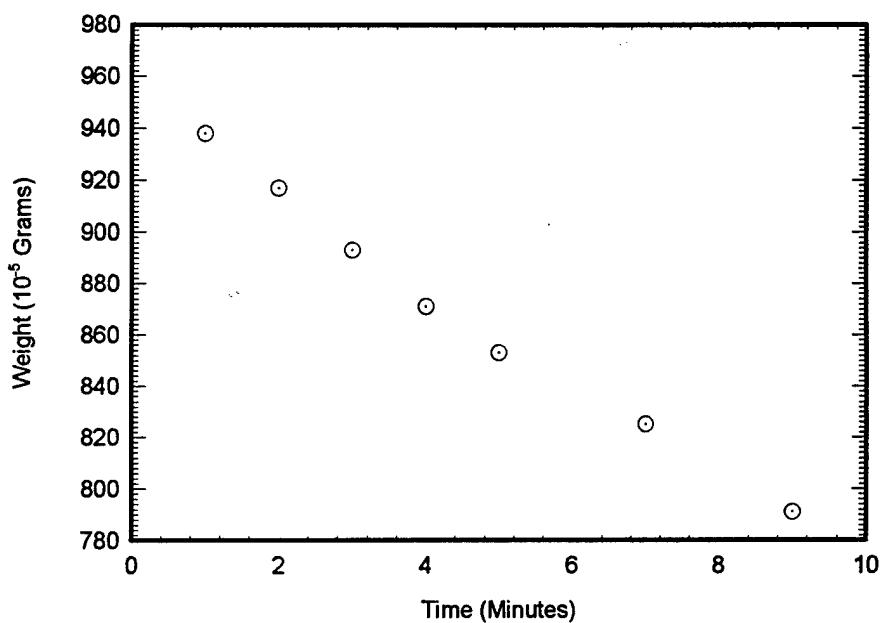
Figure 38. Moisture Loss in 80 Percent RH Equilibrated G10 Composite Powder at 23°C and 43 Percent RH (Sample 2).
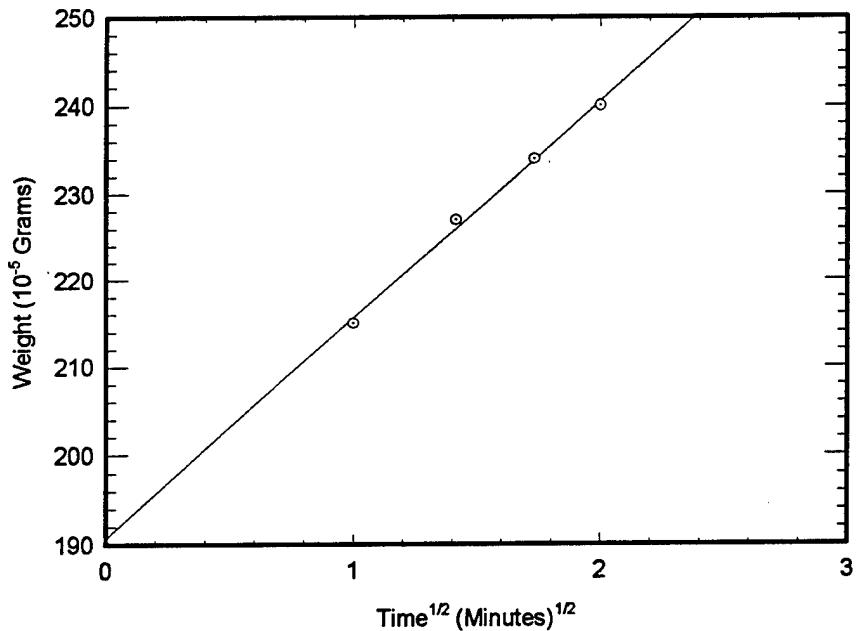
Figure 39. Moisture Gain in Dried Thin Sheet G10 Composite at 23°C
and 43 Percent RH (Sample 1).
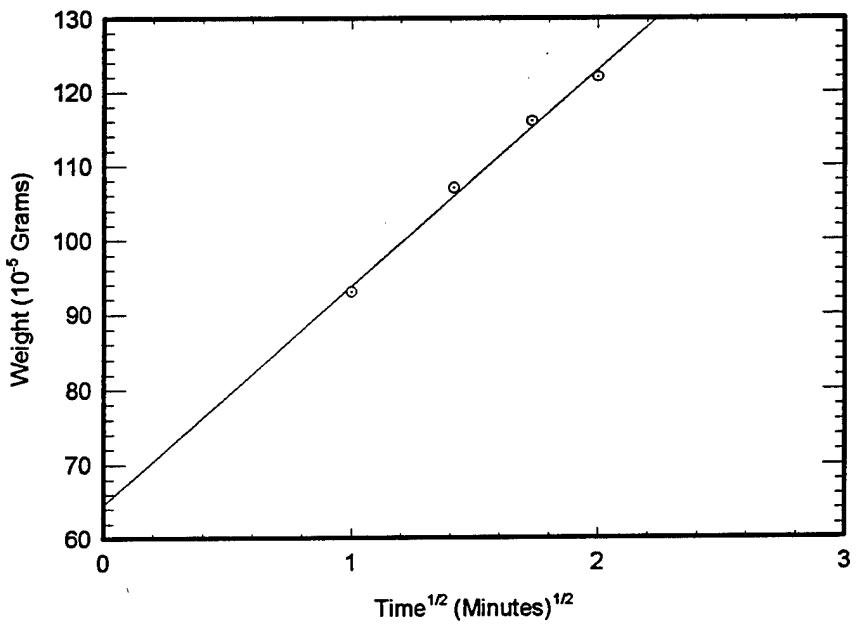
Figure 40. Moisture Gain in Dried Thin Sheet G10 Composite at 23°C
and 43 Percent RH (Sample 2).
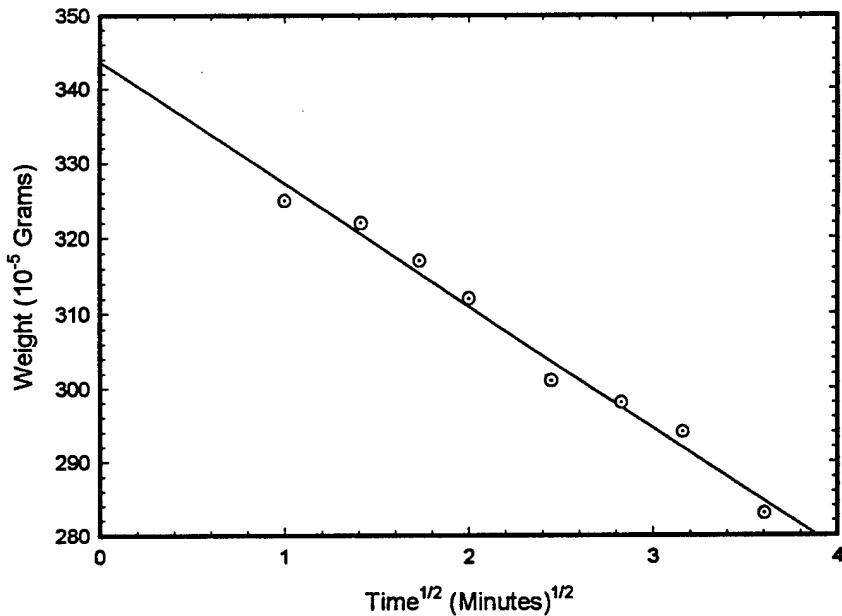
Figure 41. Moisture Loss in 80 Percent RH Equilibrated Thin Sheet G10 Composite Laminate at 23°C and 44 Percent RH (Sample 1).
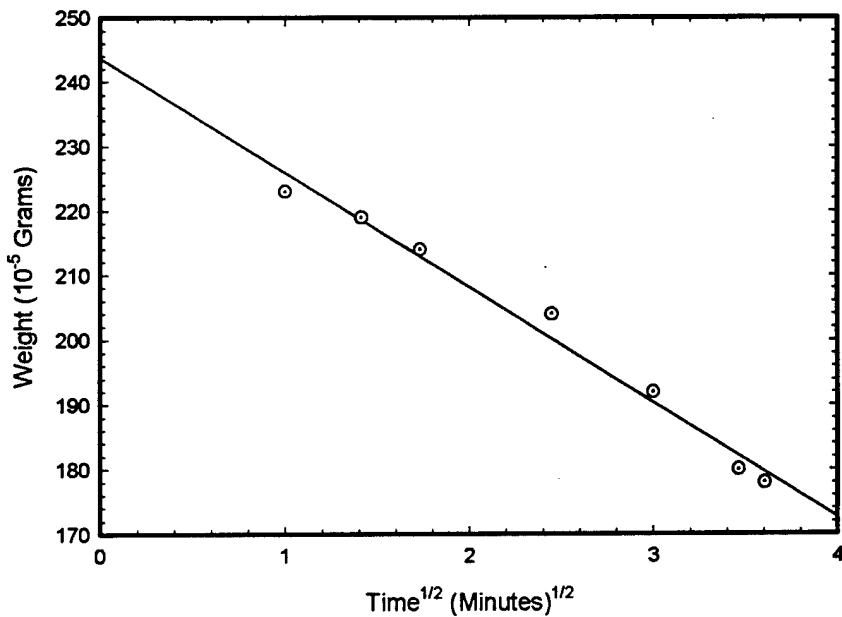
Figure 42. Moisture Loss in 80 Percent RH Equilibrated Thin Sheet G10 Composite Laminate at 23°C and 44 Percent RH (Sample 2).
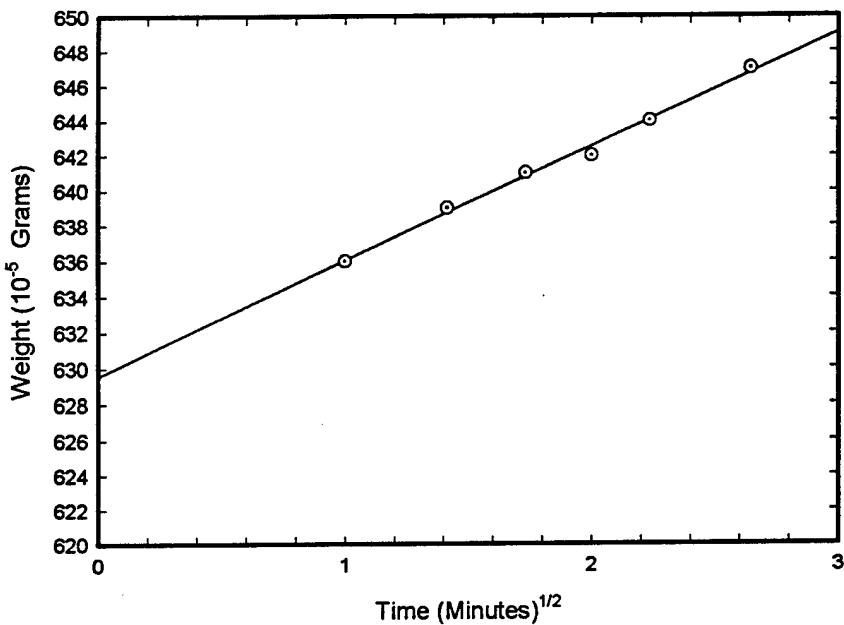
Figure 43. Moisture Gain in 23 Percent RH Equilibrated Thin Sheet
G10 Composite at 23°C and 43 RH (Sample 1).
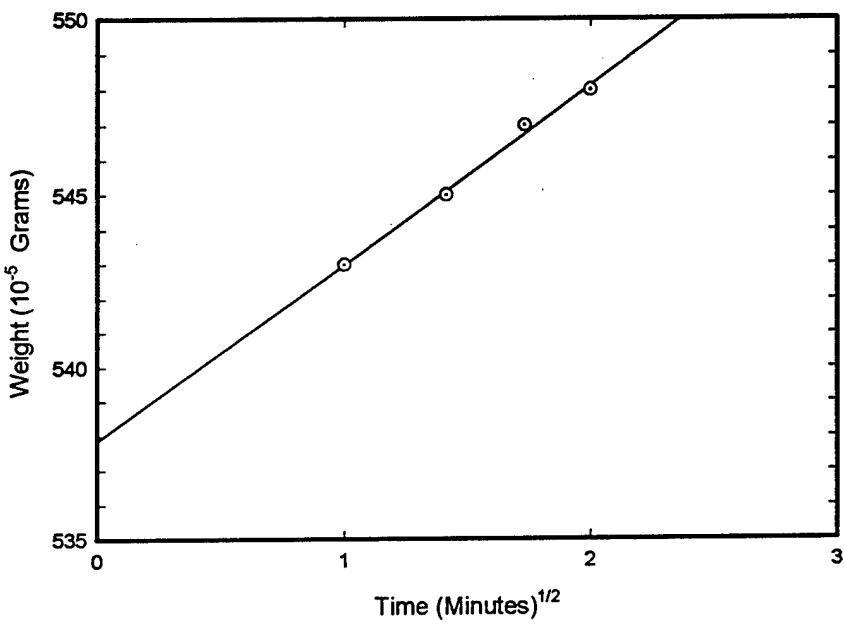
Figure 44. Moisture Gain in 23 Percent RH Equilibrated Thin Sheet
G10 Composite at 23°C and 43 RH (Sample 2).
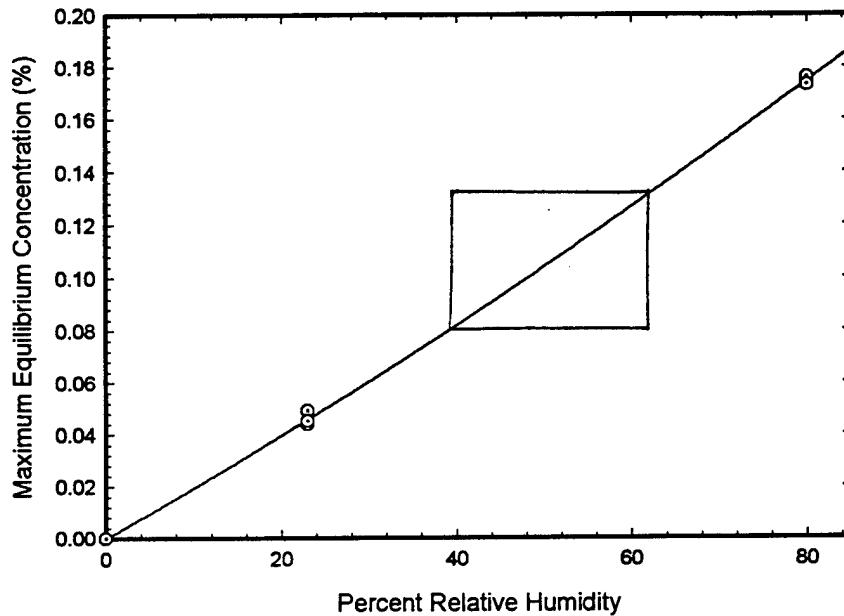
Figure 45. Maximum Moisture Equilibrium Concentration in Thin Sheet
RTM3 (Sandwich Panel) as a Function of RH.
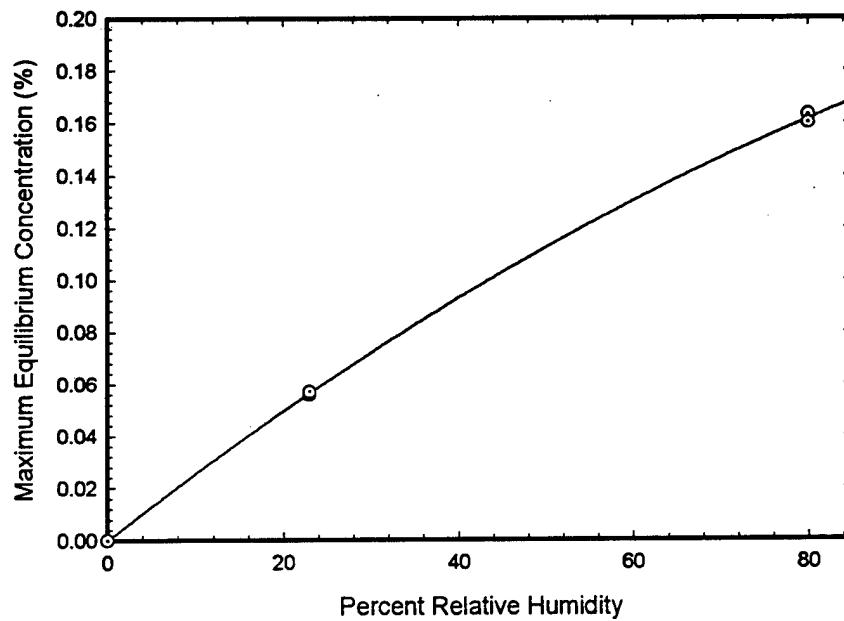
Figure 46. Maximum Moisture Equilibrium Concentration in RTM3
(Fab. 1) Thin Sheet Laminates as a Function of RH.
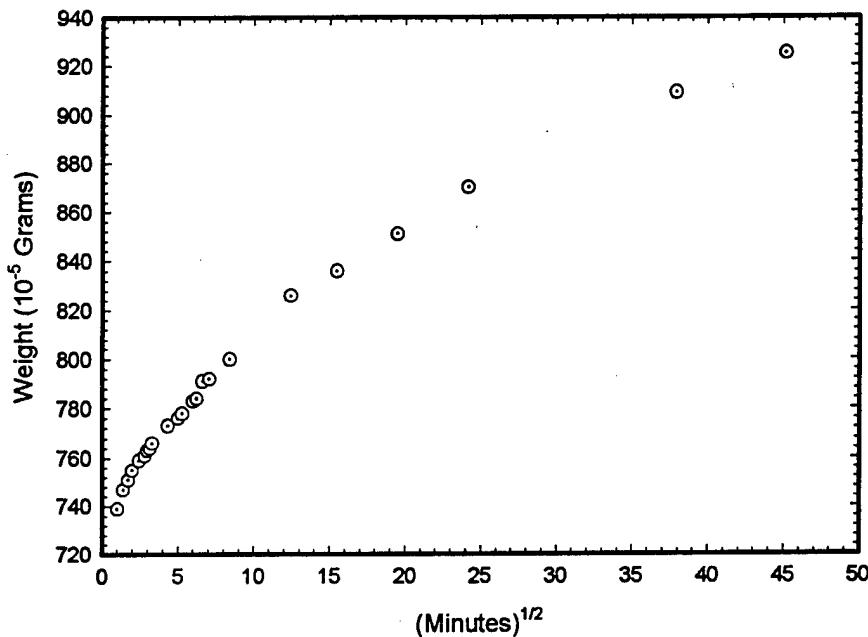
Figure 47. Moisture Sorption in Thin Sheet RTM3 (#3, dried);
run at 60 RH and 22°C
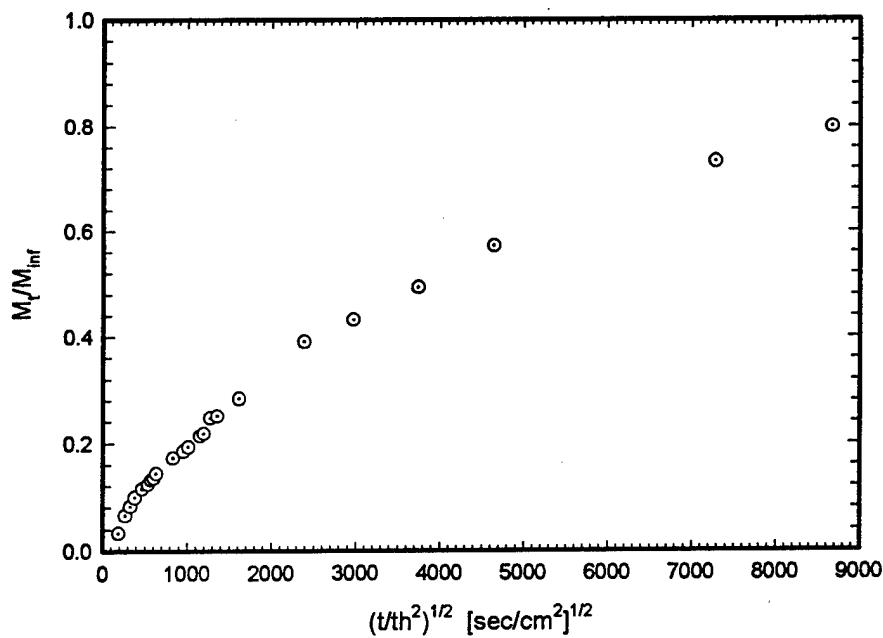
Figure 48. Reduced Sorption Data from Figure 47.
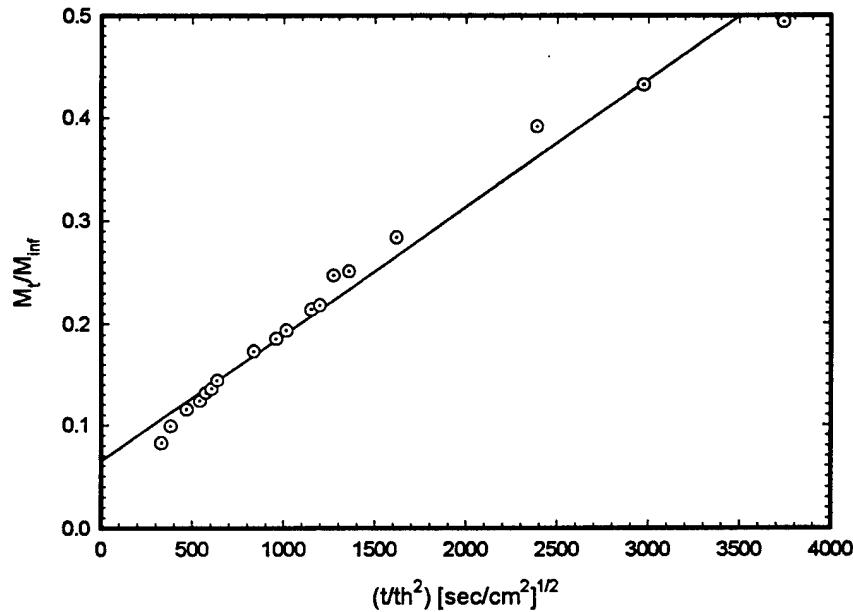
Figure 49. Magnified Initial Sorption Plot (with First Two Points Deleted).
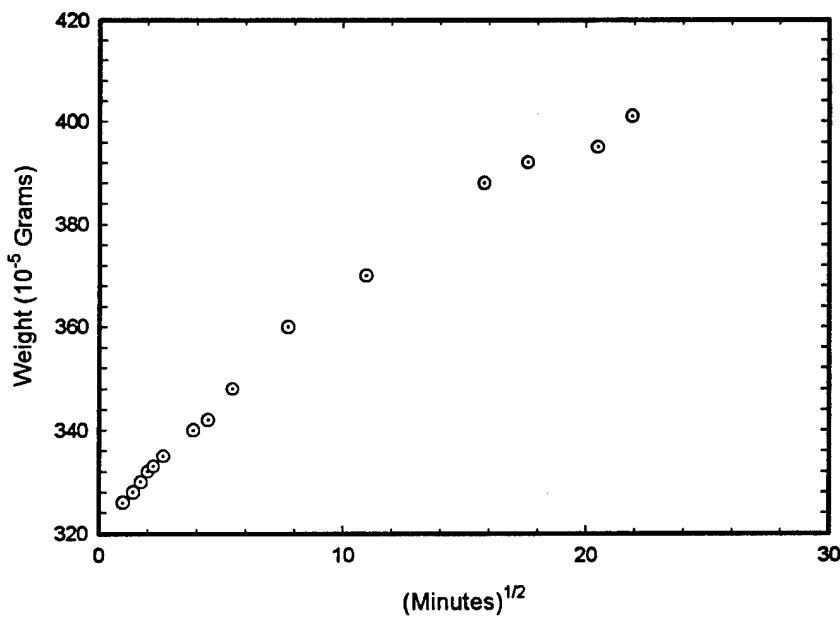
Figure 50. Moisture Sorption in Dried Thin Sheet RTM3 (Fab. 1, #1) Laminate at 43 Percent Laboratory RH and 23°C.
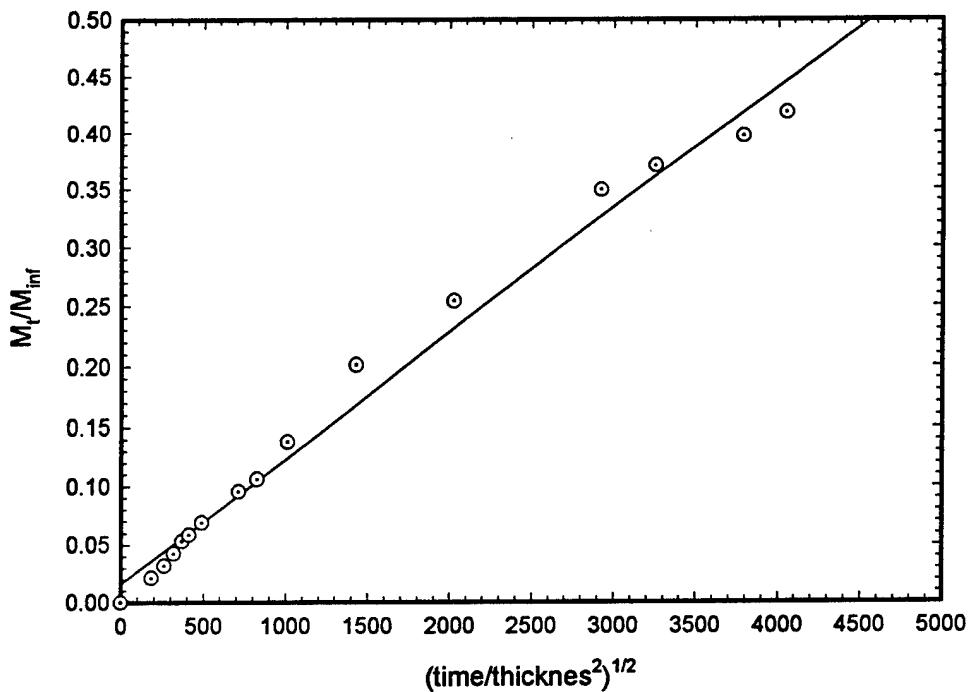
Figure 51. Least Square Fit of Initial Sorption Curve of Thin Sheet
Dried RTM3 (Fab. 1, #1) Composite Laminate at 43 Percent Lab. RH.

REFERENCES

1. Augl, J. M., *Use of Finite Element Analysis for Transient Moisture Diffusion Studies in Multilayer Composite AEM/S System Sandwich Materials*, CARDIVNSWC/TR-94/019, Nov 1994.

2. Augl, J. M. and Berger, A. E., *Moisture Diffusion Analysis in Multilayer Composite Materials by Finite Difference Analysis*, NSWCCARDIV-TR-95/013, March 1996.

3. Crank, J., *The Mathematics of Diffusion*, Clarendon Press, Oxford University, Second Editioin 1975.

4. Augl, J. M., *Sorption and Diffusion of Moisture in Multilayer Composite AEM/S System Sandwich Materials*, CARDIVNSWC/TR-94/018, Oct 1994.

5. Augl, J. M., *Moisture Diffusion in Impact Damaged Face Sheets of Composite Sandwich Materials*, NSWCCARDIV-TR-96/002, Mar 1996.

6. Van Krevelen, D. W., *Properties of Polymers*. Third Completely Revised Edition, 1990, p 574, Elsevier Publishing Co.

7. Augl, J. M. and Berger, A. E., *The Effect of Moisture on Carbon Fiber Reinforced Epoxy Composites. I Diffusion*. NSWC/WOL/TR 76-7, SEP 1976, p.39.

8. Lange, N. A., *Handbook of Chemistry*, Revised 10th Edition, McCraw-Hill Book Company, 1969 pp. 1432.

APPENDIX

This Appendix lists all pertinent experimental data developed in this investigation. The moisture equilibrium concentrations of the listed samples are obtained from the differences in the extrapolation values between the dried and the moisture equilibrated samples.

Page A-3 lists the individual data for the weight gains of dried powder samples, and the weight losses of the same set of samples after they were equilibrated at 80 percent RH. These measurements were carried out in the laboratory atmosphere (which was 23°C and 43 percent RH). The solubilities and diffusion coefficients of the undamaged RTM3 specimens, from which the powders were prepared, had been previously measured and reported in Reference 5. The times at which the weights were measured are given in minutes. The corresponding weights list only the last three significant digits (in units of 10^{-5} grams), while the extrapolated weights to time zero (in columns 5 and 10) list the full weight, including the crucible weight.

Four thin sheet laminates were prepared from impact panels (RTM3; #1 to #4). The weight gains and losses are listed on page A-4. Again, only the last two or three significant digits are listed for the weights. The extrapolated weights are given in full for the plate samples. Also listed on page A-4 are the sorption data of the four RTM3 samples after equilibration at 23 percent RH. Similar results for two thin sheet RTM3 (Fab. 1) laminates, two G10 epoxy laminate powders and two thin sheet G10 epoxy laminates are given on pages A-5 and A-6.

At the bottom half of A-6, we give two examples for preliminary estimates of diffusion coefficients from thin sheet laminate specimens where the weight gains were only continued until M_t/M_∞ exceeded 0.5 of the expected laboratory equilibrium conditions, that is, the maximum moisture concentration corresponding the laboratory RH's (which were 60 and 43 percent respectively).

On the top half of page A-7 we present a table that lists the times (in hours) it takes for given diffusion coefficients (listed in the first column) to sorb the amount of moisture to reach $M_t/M_\infty = 0.5$ (for various plate thicknesses). On the bottom half of page A-7 we list the times to reach the maximum equilibrium moisture concentration (i.e. $M_t/M_\infty = 0.993$) for thin plate samples.

Moisture Sorption in RTM3 Powders at 23 Deg.C Equilibrated at 0 and 80 Percent RH

Absorption from 0% RH					Desorption from 80% RH				
Spec.ID	Figure #	Time	Weight	Extrapol.	Spec.ID	Figure #	Time	Weight	Extrapol.
RTM3 #1	5	1	582	5.01566	RTM3#1	11	1	762	5.0178
		2	599				2	744	
		3	615				3	731	
		4	627				4	722	
		5	633				5	714	
		6	638				6	708	
		7	640				7	704	
		8	642				10	690	
		12	647						
RTM3#2	6	1	177	5.22168	RTM3#2	12	1	404	5.22422
		2	186				2	386	
		3	195				3	372	
		4	204				4	361	
		5	210				5	351	
		6	216				6	341	
		7	220				9	314	
		8	223				10	309	
		10	227						
RTM3#3	7	1	701	5.02688	RTM3#3	13	1	888	5.02902
		2	714				2	874	
		3	723				3	862	
		4	729				4	852	
		5	734				5	842	
		7	741				6	833	
		9	746				12	786	
RTM3#4	8	1	137	5.11126	RTM3#4	14	1	339	5.1135
		2	148				2	328	
		3	156				3	318	
		4	163				4	307	
		5	168				5	294	
		6	172				7	272	
		7	175				9	252	
		8	178				10	245	
RTM3#5	9	1	534	5.13522	RTM#5	15	1	734	5.13748
		2	546				2	722	
		3	556				3	707	
		4	563				4	694	
		5	568				5	682	
		8	576				7	672	
		10	581				10	666	
RTM3#6	10	1	836	4.9082	RTM#6	16	1	1035	4.91048
		2	852				2	1022	
		3	863				3	1009	
		4	872				4	997	
		5	879				5	986	
		6	884				6	976	
		8	892				9	955	
		10	900				11	947	

Moisture Sorption in RTM3 Thin Sheets at 23 Deg.C; Equilibrated at 0 and 80 Percent RH.

Absorption form 0% RH Equilibration						Desorption from 80% RH Equilibration					
Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.	Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.
RTM3#1	17	1	1	321	1.89312	RTM3#1	21	1	1	631	1.89642
		2	1.414214	327				2	1.414214	628	
		3	1.732051	330				3	1.732051	624	
		4	2	333				4	2	621	
		6	2.44949	337				5	2.236068	618	
RTM3#2	18	1	1	820	1.9281	RTM3#2	22	1	1	140	1.93147
		2	1.414214	824				2	1.414214	138	
		3	1.732051	827				3	1.732051	136	
		4	2	830				4	2	134	
		5	2.236068	832				6	2.44949	130	
RTM3#3	19	1	1	738	1.91731	RTM3#3	23	1	1	61	1.92069
		2	1.414214	741				2	1.414214	58	
		3	1.732051	743				3	1.732051	56	
		4	2	746				4	2	54	
		5	2.236068	747				6	2.44949	50	
		6	2.44949	749				7	2.645751	48	
		7	2.645751	750							
RTM3#4	20	1	1	306	1.75294	RTM3#4	24	1	1	588	1.75597
		2	1.414214	310				2	1.414214	585	
		3	1.732051	315				3	1.732051	582	
		4	2	318				4	2	579	
		5	2.236068	320							

Moisture Sorption in RTM3 Thin Sheets at 23 Deg.C; Equilibrated at 23 Percent RH.

Absorption from 23% Equilibration						Absorption from 23% RH Equilibration					
Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.	Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.
RTM3#1	25	1	1	407	1.89396	RTM3#2	26	1	1	910	1.92904
		2	1.414214	411				2	1.414214	912	
		3	1.732051	414				3	1.732051	915	
		4	2	417				4	2	917	
		5	2.236068	421				7	2.645751	920	
		6	2.44949	424							
		8	2.828427	427							
		9	3	428							
RTM3#3	27	1	1	826	1.91731	RTM3#4	28	1	1	382	1.75373
		2	1.414214	829				2	1.414214	385	
		3	1.732051	831				3	1.732051	387	
		4	2	833				5	2.236068	393	
		6	2.44949	837				6	2.44949	394	
		7	2.645751	839							
		8	2.828427	840							

Moisture Sorption in RTM3 (Fab. 1) Thin Sheet Composite; Equilibrated at 0 and 80 Percent RH

Absorption from 0% Equilibration						Desorption from 80% Equilibration					
Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.	Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.
RTM3-1#1	29	1	1	326	1.91322	RTM3-1#1	31	1	1	628	1.91633
		2	1.414214	328				2	1.414214	624	
		3	1.732051	330				3	1.732051	621	
		4	2	332				4	2	617	
		5	2.236068	333				5	2.236068	615	
		7	2.645751	335				8	2.828427	613	
		15	3.872983	340				11	3.162278	609	
								16	4	605	
								18	4.242641	603	
RTM3-1#2	30	1	1	713	1.91708	RTM3-1#2	32	1	1	2007	1.92015
		2	1.414214	717				2	1.414214	2004	
		3	1.732051	720				3	1.732051	2000	
		4	2	722				4	2	1996	
		5	2.236068	723				5	2.236068	1994	
		8	2.828427	725				7	2.645751	1991	
		11	3.162278	728				13	3.605551	1984	
		12	3.464102	730				16	4	1980	
		14	3.741657	733				18	4.242641	1978	

Moisture Sorption in RTM3 (Fab. 1) Thin Sheet Composite; Equilibrated at 23 Percent RH

Absorption from 23% Equilibration						Absorption from 23% Equilibration					
Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.	Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.
RTM3-1#1	33	1	1	433	1.91429	TRM3-1#2	34	1	1	822	1.91817
		2	1.414214	434				2	1.414214	824	
		3	1.732051	435				3	1.732051	826	
		5	2.236068	437				4	2	827	
		6	2.44949	438				6	2.44949	829	

Moisture Sorption in G10 Composite Powder at 23 Deg.C; Equilibrated at 0 and 80 Percent RH.

Absorption from 0% Equilibration						Absorption from 80% Equilibration					
Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.	Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.
G10 #1	35	1		38	5.16436	G10 #1	37	1		7333	5.30014
		2		62				2		7310	
		3		77				3		7286	
		4		94				4		7265	
		5		106				5		7251	
		7		137				7		7216	
		8		147				9		7191	
		10		166				12		7151	
G10#2	36	1		456	5.17356	G10 #2	38	1		938	5.30963
		2		475				2		917	
		3		495				3		893	
		4		513				4		871	
		5		528				5		853	
		7		560				7		825	
		8		574				9		791	

Moisture Sorption in G10 Thin Sheet Composite; Equilibrated at 0, 80, and 23 Percent RH.

Absorption from 0 RH						Desorption from 80 Percent RH					
Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.	Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.
G10 #1	39	1	1	215	1.55191	G10 #1	41	1	1	244	1.56262
		2	1.414214	227				2	1.414214	236	
		3	1.732051	234				3	1.732051	230	
		4	2	240				4	2	226	
G10 #2	40	1	1	93	1.58065		42	1	1	159	1.59179
		2	1.414214	107				2	1.414214	151	
		3	1.732051	116				3	1.732051	144	
		4	2	122				4	2	139	

Absorption in Sample Equilibrated at 23 Percent RH.

Absorption from 0 RH					
Spec. ID	Figure #	Minutes	Sqrt(Min)	Weight	Extrapol.
G10 #1	43	1	1	636	1.55630
		2	1.414214	639	
		3	1.732051	641	
		4	2	642	
		5	2.236068	644	
		7	2.645751	647	
G10#2	44	1	1	526	1.58521
		2	1.414214	528	
		3	1.732051	529	
		5	2.236068	531	
		6	2.44949	533	
		7	2.645751	534	

Diffusion Coefficient from Thin Sheet RTM3 Laminates

Absorption of dried sample at 60 RH and 23 Deg.C					
Spec. ID	Figure #	Minutes	(t/th^2)^.5	Weight	Mt/Minf
RTM3 #3	47	1	191.998	739	0.032922
		2	271.5262	747	0.065844
		3	332.5503	751	0.082305
		4	383.996	755	0.098765
		6	470.2971	759	0.115226
		8	543.0523	761	0.123457
		9	575.994	763	0.131687
		10	607.1509	764	0.135802
		11	636.7853	766	0.144033
		19	836.8998	773	0.17284
		25	959.9899	776	0.185185
		28	1015.958	778	0.193416
		36	1151.988	783	0.213992
		39	1199.027	784	0.218107
		44	1273.571	791	0.246914
		50	1357.631	792	0.251029
		71	1617.804	800	0.283951
		155	2390.356	826	0.390947
		240	2974.42	836	0.432099
		380	3742.73	851	0.493827
		585	4643.812	870	0.572016
		1440	7285.811	909	0.73251
		2040	8671.85	925	0.798354

Diffusion Coefficient from Thin Sheet RTM3 (Fab. 1) Lam.

Absorption of dried sample at 43 Lab.RH and 23 Deg.C					
Spec. ID	Figure #	Minutes	(t/th^2)^.5	Weight	Mt/Minf
RTM3 #1	51	1	184.8679	326	0.021164
		2	261.4427	328	0.031746
		3	320.2007	330	0.042328
		4	369.7359	332	0.05291
		5	413.3773	333	0.058201
		7	489.1146	335	0.068783
		15	715.9905	340	0.095238
		20	826.7546	342	0.10582
		30	1012.563	348	0.137566
		60	1431.981	360	0.201058
		120	2025.127	370	0.253968
		250	2923.019	388	0.349206
		310	3254.936	392	0.37037
		420	3788.665	395	0.386243
		480	4050.254	401	0.417989
		4293	12112.73	447	0.661376

Times (in hours) for Plate Specimens to sorb 0.5 Mt/Minf for given Diffusion Coefficients (Thickness in cm)											
Thickness	0.08	0.09	0.1	0.11	0.12	0.13	0.14	0.15	0.16	0.17	0.18	0.19
Diff.Coeff.												
4.00E-10	217.7778	275.6250	340.2778	411.7361	490.0000	575.0694	666.9444	765.6250	871.1111	983.4028	1102.500	1228.403
5.00E-10	174.2222	220.5000	272.2222	329.3889	392.0000	460.0556	533.5556	612.5000	696.8889	786.7222	882.0000	982.7222
6.00E-10	145.1852	183.7500	226.8519	274.4907	326.6667	383.3796	444.6296	510.4167	580.7407	655.6019	735.0000	818.9352
7.00E-10	124.4444	157.5000	194.4444	235.2778	280.0000	328.6111	381.1111	437.5000	497.7778	561.9444	630.0000	701.9444
8.00E-10	108.8889	137.8125	170.1389	205.8681	245.0000	287.5347	333.4722	382.8125	435.5556	491.7014	551.2500	614.2014
9.00E-10	96.7901	122.5000	151.2346	182.9938	217.7778	255.5864	296.4198	340.2778	387.1605	437.0679	490.0000	545.9568
1.00E-09	87.1111	110.2500	136.1111	164.6944	196.0000	230.0278	266.7778	306.2500	348.4444	393.3611	441.0000	491.3611
2.00E-09	43.5556	55.1250	68.0556	82.3472	98.0000	115.0139	133.3889	153.1250	174.2222	196.6806	220.5000	245.6806
3.00E-09	29.0370	36.7500	45.3704	54.8981	65.3333	76.6759	88.9259	102.0833	116.1481	131.1204	147.0000	163.7870
4.00E-09	21.7778	27.5625	34.0278	41.1736	49.0000	57.5069	66.6944	76.5625	87.1111	98.3403	110.2500	122.8403
5.00E-09	17.4222	22.0500	27.2222	32.9389	39.2000	46.0056	53.3556	61.2500	69.6889	78.6722	88.2000	98.2722
6.00E-09	14.5185	18.3750	22.6852	27.4491	32.6667	38.3380	44.4630	51.0417	58.0741	65.5602	73.5000	81.8935
7.00E-09	12.4444	15.7500	19.4444	23.5278	28.0000	32.8611	38.1111	43.7500	49.7778	56.1944	63.0000	70.1944
8.00E-09	10.8889	13.7813	17.0139	20.5868	24.5000	28.7535	33.3472	38.2813	43.5556	49.1701	55.1250	61.4201
9.00E-09	9.6790	12.2500	15.1235	18.2994	21.7778	25.5586	29.6420	34.0278	38.7160	43.7068	49.0000	54.5957
1.00E-08	8.7111	11.0250	13.6111	16.4694	19.6000	23.0028	26.6778	30.6250	34.8444	39.3361	44.1000	49.1361
2.00E-08	4.3556	5.5125	6.8056	8.2347	9.8000	11.5014	13.3389	15.3125	17.4222	19.6681	22.0500	24.5681
3.00E-08	2.9037	3.6750	4.5370	5.4898	6.5333	7.6676	8.8926	10.2083	11.6148	13.1120	14.7000	16.3787
4.00E-08	2.1778	2.7563	3.4028	4.1174	4.9000	5.7507	6.6694	7.6563	8.7111	9.8340	11.0250	12.2840
5.00E-08	1.7422	2.2050	2.7222	3.2939	3.9200	4.6006	5.3356	6.1250	6.9689	7.8672	8.8200	9.8272
6.00E-08	1.4519	1.8375	2.2685	2.7449	3.2667	3.8338	4.4463	5.1042	5.8074	6.5560	7.3500	8.1894
7.00E-08	1.2444	1.5750	1.9444	2.3528	2.8000	3.2861	3.8111	4.3750	4.9778	5.6194	6.3000	7.0194
8.00E-08	1.0889	1.3781	1.7014	2.0587	2.4500	2.8753	3.3347	3.8281	4.3556	4.9170	5.5125	6.1420
9.00E-08	0.9679	1.2250	1.5123	1.8299	2.1778	2.5559	2.9642	3.4028	3.8716	4.3707	4.9000	5.4596
1.00E-07	0.8711	1.1025	1.3611	1.6469	1.9600	2.3003	2.6678	3.0625	3.4844	3.9336	4.4100	4.9136

Hours to saturate thin plates to 99.3% of the solubility with moisture. (Thickness: in inches and cm)											
Thick.inch	0.009	0.01	0.011	0.012	0.013	0.014	0.015	0.016	0.017	0.018	0.019	0.02
cm	0.02286	0.0254	0.02794	0.03048	0.03302	0.03556	0.0381	0.04064	0.04318	0.04572	0.04826	0.0508
4.00E-10	177.8222	219.5336	265.6357	316.1284	371.0118	430.2859	493.9506	562.0060	634.4521	711.2889	792.5163	878.1344
5.00E-10	142.2578	175.6269	212.5085	252.9027	296.8094	344.2287	395.1605	449.6048	507.5617	569.0311	634.0131	702.5076
6.00E-10	118.5482	146.3557	177.0904	210.7523	247.3412	286.8573	329.3004	374.6707	422.9681	474.1926	528.3442	585.4230
7.00E-10	101.6127	125.4478	151.7918	180.6448	212.0067	245.8776	282.2575	321.1463	362.5441	406.4508	452.8665	501.7911
8.00E-10	88.9111	109.7668	132.8178	158.0642	185.5059	215.1429	246.9753	281.0030	317.2261	355.6445	396.2582	439.0672
9.00E-10	79.0321	97.5705	118.0603	140.5015	164.8941	191.2382	219.5336	249.7805	281.9787	316.1284	352.2295	390.2820
1.00E-09	71.1289	87.8134	106.2543	126.4514	148.4047	172.1144	197.5803	224.8024	253.7809	284.5156	317.0065	351.2538
2.00E-09	35.5644	43.9067	53.1271	63.2257	74.2024	86.0572	98.7901	112.4012	126.8904	142.2578	158.5033	175.6269
3.00E-09	23.7096	29.2711	35.4181	42.1505	49.4682	57.3715	65.8601	74.9341	84.5936	94.8385	105.6688	117.0846
4.00E-09	17.7822	21.9534	26.5636	31.6128	37.1012	43.0286	49.3951	56.2006	63.4452	71.1289	79.2516	87.8134
5.00E-09	14.2258	17.5627	21.2509	25.2903	29.6809	34.4229	39.5161	44.9605	50.7562	56.9031	63.4013	70.2508
6.00E-09	11.8548	14.6356	17.7090	21.0752	24.7341	28.6857	32.9300	37.4671	42.2968	47.4193	52.8344	58.5423
7.00E-09	10.1613	12.5448	15.1792	18.0645	21.2007	24.5878	28.2258	32.1146	36.2544	40.6451	45.2866	50.1791
8.00E-09	8.8911	10.9767	13.2818	15.8064	18.5506	21.5143	24.6975	28.1003	31.7226	35.5644	39.6258	43.9067
9.00E-09	7.9032	9.7570	11.8060	14.0502	16.4894	19.1238	21.9534	24.9780	28.1979	31.6128	35.2229	39.0282
1.00E-08	7.1129	8.7813	10.6254	12.6451	14.8405	17.2114	19.7580	22.4802	25.3781	28.4516	31.7007	35.1254
2.00E-08	3.5564	4.3907	5.3127	6.3226	7.4202	8.6057	9.8790	11.2401	12.6890	14.2258	15.8503	17.5627
3.00E-08	2.3710	2.9271	3.5418	4.2150	4.9468	5.7371	6.5860	7.4934	8.4594	9.4839	10.5669	11.7085
4.00E-08	1.7782	2.1953	2.6564	3.1613	3.7101	4.3029	4.9395	5.6201	6.3445	7.1129	7.9252	8.7813
5.00E-08	1.4226	1.7563	2.1251	2.5290	2.9681	3.4423	3.9516	4.4960	5.0756	5.6903	6.3401	7.0251
6.00E-08	1.1855	1.4636	1.7709	2.1075	2.4734	2.8686	3.2930	3.7467	4.2297	4.7419	5.2834	5.8542
7.00E-08	1.0161	1.2545	1.5179	1.8064	2.1201	2.4588	2.8226	3.2115	3.6254	4.0645	4.5287	5.0179
8.00E-08	0.8891	1.0977	1.3282	1.5806	1.8551	2.1514	2.4698	2.8100	3.1723	3.5564	3.9626	4.3907
9.00E-08	0.7903	0.9757	1.1806	1.4050	1.6489	1.9124	2.1953	2.4978	2.8198	3.1613	3.5223	3.9028
1.00E-07	0.7113	0.8781	1.0625	1.2645	1.4840	1.7211	1.9758	2.2480	2.5378	2.8452	3.1701	3.5125

DISTRIBUTION

	Copies		Copies

DOD ACTIVITIES (CONUS)

ATTN 332 (JAMES J. KELLY) 1
    331PO (DR K J WYNNE) 1
DIRECTOR
OFFICE OF NAVAL RESEARCH
BALLSTON TOWER - 504
800 N QUINCY STREET
ARLINGTON VA 22217-5660

ATTN AIR-52032 (S STEEL) 1
    AIR-52032 (D MOORE) 1
COMMANDER
NAVAL AIR SYSTEMS COMMAND
WASHINGTON DC 20362

ATTN SEA-03B 1
    SEA-03R (SPENCER) 1
    SEA-05R 1
    SEA-062D 1
    SEA-06K42 (EADIE) 1
    SEA-09G32 1
COMMANDER
NAVAL SEA SYSTEMS COMMAND
WASHINGTON DC 20362

ATTN SP2731 (L THROCKMORTON) 1
    SP2731 (K KLABER) 1
STRATEGIC SYSTEM PROJECT OFFICE
WASHINGTON DC 20376

ATTN 6380 (DR R BADALIANCE) 1
DIRECTOR
NAVAL RESEARCH LABORATORY
WASHINGTON DC 20375

ATTN 824 (LOGAN) 1
COMMANDER
NAVAL COMMAND, CONTROL, AND OCEAN
    SURVEILLANCE CENTER
53560 HULL ST
SAN DIEGO CA 92152-5001

ATTN B44 (A E BERGER) 1
    F24 (CAMPBELL) 1
COMMANDER
NAVAL SURFACE WARFARE CENTER
DAHLGREN DIVISION
17320 DAHLGREN RD
DAHLGREN VA 22448-5100

DIRECTOR OF DEVELOPMENT
ARMY MATERIAL COMMAND
GRAVELEY POINT
WASHINGTON DC 20316 1

ATTN WL/DOC (TECH LIBRARY) 1
    WL/MLBC (C BROWNING) 1
WRIGHT LABORATORY
WRIGHT PATTERSON AFB OH 45433-7411

DEFENSE TECHNICAL INFORMATION CENTER
8725 JOHN J KINGMAN ROAD
SUITE 09844
FORT BELVOIR VA 22060-6218 2

NON-DOD ACTIVITIES

ATTN T L ST CLAIR 1
MAIL STOP 226
NASA LANGLEY RESEARCH CENTER
HAMPTON VA 23665

DISTRIBUTION (Continued)

		Copies			Copies
NSWC CARDEROCK DIVISION			643	(SORATHIA)	1
INTERNAL DISTRIBUTION			65	(BEACH)	1
			6501	(BENSON)	1
011	(CORRADO)	1	655	(CRITCHFIELD)	1
0112	(DOUGLAS)	1	6551	(CAMPONESCHI)	1
0115	(CAPLAN)	1	6551	(MAYES)	1
0119	(MESSICK)	1	6552	(GIPPLE)	1
3021	(COLEGROVE)	1	6552	(LOUP)	1
3023	(TIC/CARDEROCK)	1	6552	(TELEGADAS)	1
3023	(TIC/WHITE OAK)	1	6553	(COFFIN)	1
3024	(TIC/ANNAPOLIS)	1	6553	(CRANE)	1
60	(WACKER)	1	6553	(JUSKA)	1
601	(MORTON)	1	6553	(WILLIAMS)	2
602	(ROCKWELL)	1	68	(MUELLER)	1
603	(CAVALLARO)	1	681	(AUGL)	2
61	(HOLSBERG)	1	681	(FEDDERLY)	1
64	(FISCHER)	1	681	(HARTMANN)	1
6401	(CASTELLI)	1	681	(J D LEE)	1
641	(HOWELL)	1	823	(WILHELMI)	1